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The Journal
of the
SOCIETY OF DYERS AND COLOURISTS

Volume 72 Number 3

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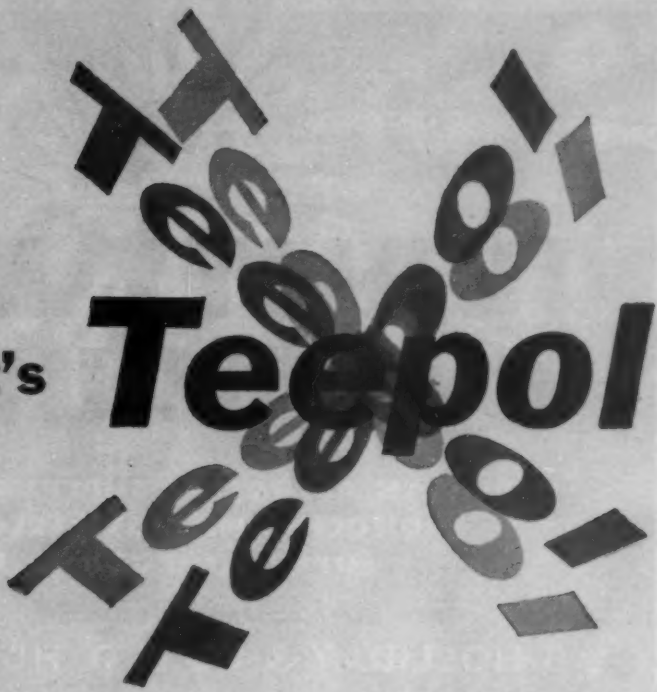

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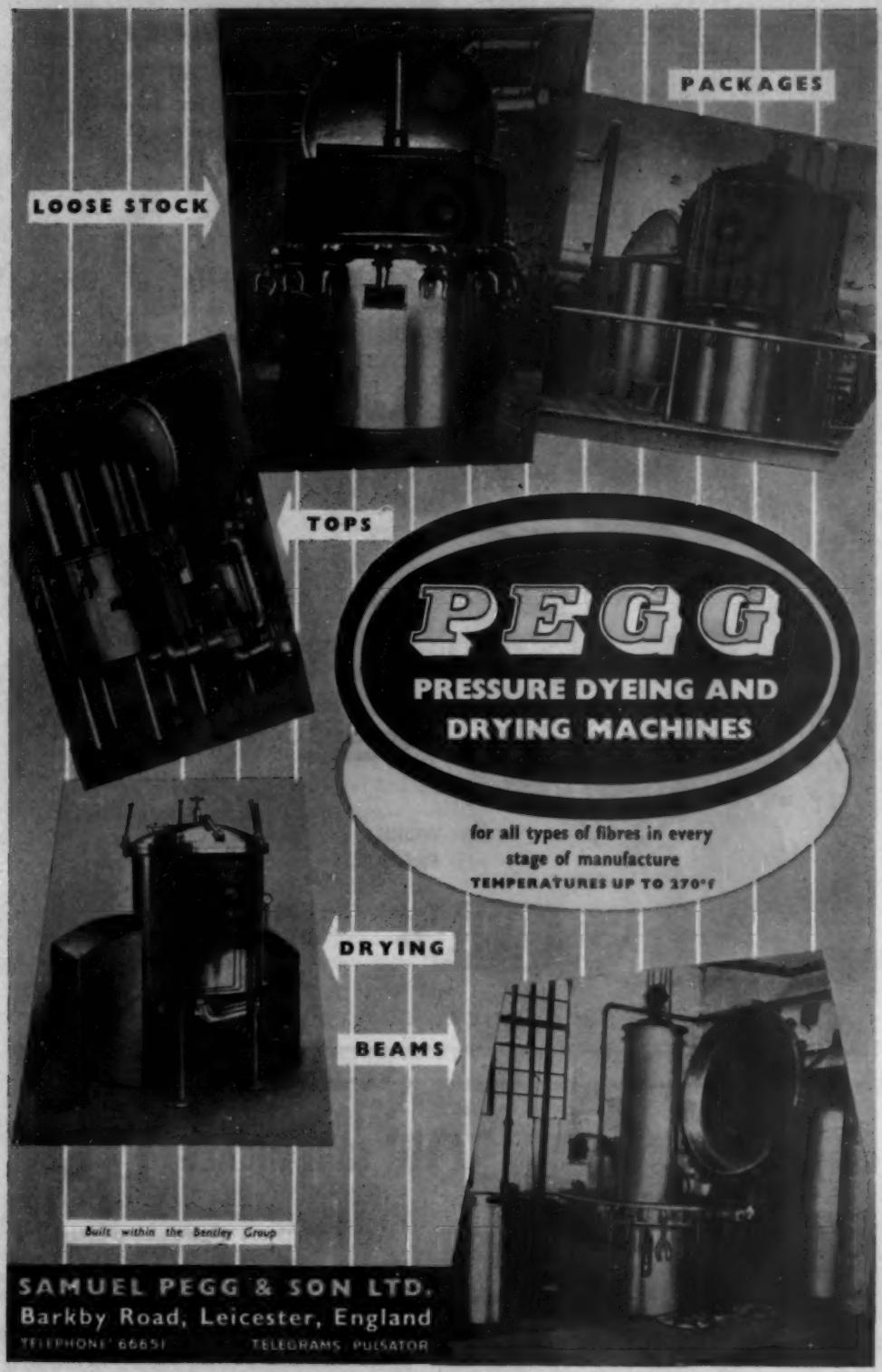
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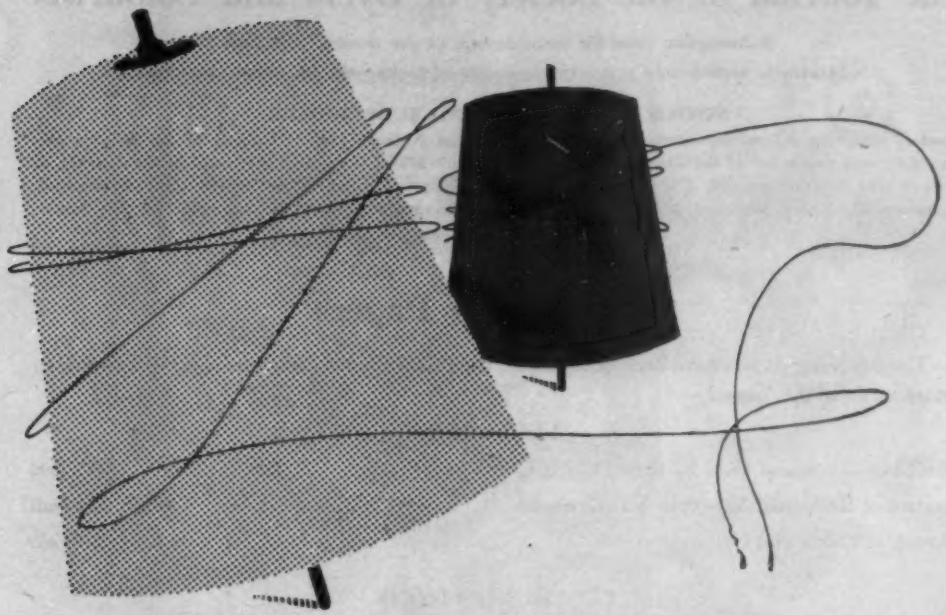

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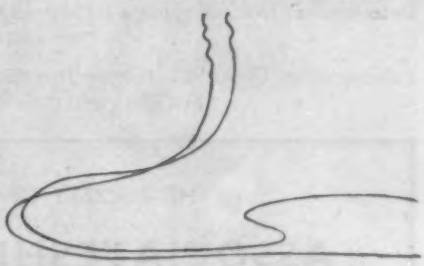

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1956 and pages 369–373 of the July 1955 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, Dean House, 19 Piccadilly, Bradford 1, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal* —

LECTURES

The Classification of Dyes by their Dyeing Characteristics	*W. Beal*
Control of Reducing Agents in Vat Dyebaths	*W. J. Marshall*
Dyeing of Orlon and Orlon Mixtures	*I. M. S. Walls*

COMMUNICATIONS

Package Dyeing—II	*W. Armfield, J. Boulton, and J. Crank*
The Rotproofing of Viscose Rayon—II	*J. W. Bell and M. M. Ramsey*
The Polarography of Azo Dyes	*J. de O. Cabral and H. A. Turner*
Interaction between Chlorazol Sky Blue FF and Chrysophenine G in Aqueous Solution	*A. N. Derbyshire and R. H. Peters*
Identification of Vat Dyes	*D. A. Derrett-Smith and J. Gray*
Detection of Dyes possessing a *p*-Phenylenediamine or a *p*-Nitroaniline Structure	*F. Feigl and C. C. Neto*
Polarographic Contribution to an Investigation of Fur Dyes and their Oxidation Products	*G. Sandberg*

THE SOCIETY OF DYERS AND COLOURISTS

ASSOCIATESHIP EXAMINATIONS 1956

The next Associateship Examinations will be held on **Thursday, Friday and Saturday, 14, 15 and 16 June 1956.**
Closing date for receipt of candidates' applications **31 March 1956**

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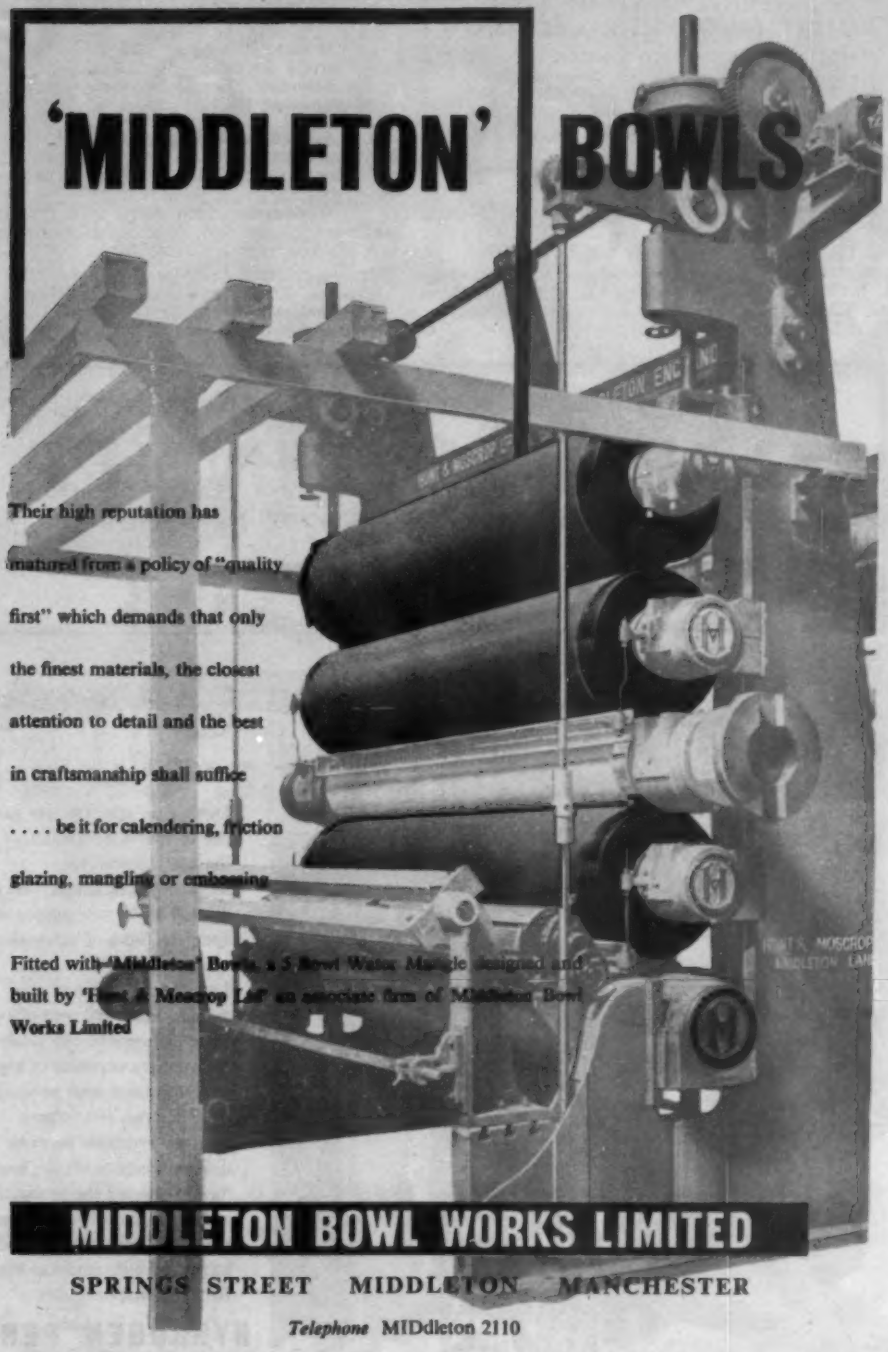

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FORTHCOMING MEETINGS OF THE SOCIETY

POSTPONEMENT OF THE FOURTH LONDON LECTURE

Owing to illness, the Lecture *Colour in Fashion* which was to have been given by James Laver, Esq, C.B.E, on Friday, April 20th, at the Waldorf Hotel, London, has been postponed until October 1956.

The date will be announced later

Saturday, 24th March 1956

NORTHERN IRELAND SECTION. Annual General Meeting and Dinner. Details later.

Tuesday, 10th April 1956

SCOTTISH SECTION. *Textile Invention and the Dry Cleaner.* A. J. Crockatt, Esq., M.Sc., F.R.I.C. (Eastmans Dyers and Cleaners Ltd.). St. Enoch's Hotel, **Glasgow.** 7 p.m.

Friday, 13th April 1956

LONDON SECTION. Annual General Meeting and Dinner. Aldwych Brasserie, **London, W.C.2.** 6.30 p.m.

Friday, 20th April 1956

MANCHESTER SECTION. *The Mechanism of Jigger Dyeing.* Dr. Ashworth. Textile Institute, 10 Blackfriars Street, **Manchester 10.** 6.30 p.m.

Saturday, 21st April 1956

BRADFORD JUNIOR BRANCH. Annual General Meeting. Technical College, **Bradford.** 10.15 a.m.

Wednesday, 25th April 1956

HUDDERSFIELD SECTION. Visit to Messrs. Stevensons Dyers Ltd., **Ambergate,** Derbyshire.

MIDLANDS SECTION. *A New Approach to High Temperature Dyeing.* S. N. Bradshaw, Esq. Kings Head Hotel, **Loughborough.** Preceded by the Annual General Meeting of the Section. 6.30 p.m.

THE SOCIETY OF DYERS AND COLOURISTS

ANNUAL DINNER

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The Society of Dyers and Colourists

LONDON SECTION

PERKIN CENTENARY DINNER AND BALL

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on

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at

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The Centenary will be celebrated by Lectures and Social Functions in London on

Monday 7th May

Evening 8.30	Soirée— Principal overseas guests will be entertained by representatives of
Cars 10.30	the Councils of the sponsoring bodies at Tallow Chandlers' Hall in the City of London, prior to the opening of the Conference
	Light Refreshments *Music* *Evening Dress or Dinner Jacket*

Tuesday 8th May

Afternoon 3.0	Opening paper on *The Life and Work of Perkin* will be read by Prof. John Read Ph.D M.A F.R.S
Evening 8.0	Reception for all those attending the Conference, including guests at Guildhall in the City of London
Cars 10.30	by kind permission of The Rt. Hon. The Lord Mayor of London
	Refreshments *Music* *Evening Dress or Dinner Jacket*

Wednesday 9th May

Morning 11.0	A paper on *The Development of the Dyestuffs Industry* will be read by Clifford Paine Esq B.Sc
Afternoon 3.0	A paper on *The Tinctorial Arts Today* will be read by J. G. Evans Esq M.Sc.Tech
Evening 7 *for* 7.45	**A Banquet will be held at The Dorchester, Park Lane, London** *Ballroom Entrance Evening Dress Orders and Decorations Cars 10.30*

Thursday 10th May

Morning 11.0	A paper on *The Development of Organic Chemistry since Perkin's Discovery* will be read by Sir Alexander R Todd M.A D.Phil D.Sc LL.D F.R.S
Evening 7 *for* 7.45	A Perkin Dinner and Ball will be given by the London Section of the Society of Dyers and Colourists at The Waldorf Hotel, Aldwych, London (Tickets 45s each) *Evening Dress Dancing until 1 a.m*

All the papers will be read in the theatre of The Royal Institution, Albemarle Street, London

There will be a Perkin Exhibition at the Science Museum, South Kensington, London, coincident with the celebrations and remaining open for not less than a month

The charges for those attending the Conference and other functions are
Conference Registration Fee and Guildhall Reception **£3 3 0**, Banquet **£3 3 0**
Registration *only* for lectures **£2 2 0**

There is available an inclusive charge of **£5 10 0** for Registration, Reception and Banquet but the charge for an accompanying guest (Ladies are invited) for the Banquet is **£3 3 0** and for the Guildhall Reception **£1 1 0**

Applications for tickets for Banquet, Receptions and Lectures should be sent AT ONCE to the *Perkin Centenary General Secretary at the above address* **and please state whether accompanied by a lady when attending the Banquet and Receptions**

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THE JOURNAL
OF THE
Society of Dyers and Colourists

Volume 72 Number 3 MARCH 1956 *Issued Monthly*

Proceedings of the Society

Effect of Acids, Alkalis, and Salts on the Colour of Blue Anthraquinone Acid Dyes

R. H. Peters and H. H. Sumner

Meeting of the West Riding Section held at the Victoria Hotel, Bradford, on 24th February 1955, Mr. G. B. Angus in the chair

Certain blue anthraquinone acid dyes show colour changes when the dyed material is treated with acid, alkali, or salt. For eight dyes, these properties have been correlated with the changes which occur when these reagents are added to solutions of the dyes in water. Addition of acid to the dye solution gives a salt by reaction with amino groups in the dye. This results in a reduction of the intensity of the absorption band characteristic of the blue colour and produces one at shorter wavelengths. These changes have enabled the dissociation constants to be calculated, and the latter run parallel with results of acid treatment of dyed patterns. Alkali, on the other hand, reacts with any hydroxyl groups present, giving rise to a colour change. Here it is not possible to calculate the dissociation constants, but the spectral changes in solution agree with the colour changes on the fibre. Addition of salt to solutions of these dyes does not produce a wavelength change but merely a reduction in intensity, presumably due to aggregation. This effect is in agreement with the changes which occur on salt spotting. Another factor is that the salt solution may cause desorption of the dye from the fibre, and hence migration can occur. The susceptibility of a dyed fibre to salt spotting has been shown to parallel the ability of the dye to be desorbed as measured by the affinity of the dye for the fibre.

Introduction

The choice of dyes for a particular purpose depends on many considerations, such as fastness to light and washing and the nature of the material to be dyed. Of the types of dyes available for dyeing wool, the level-dyeing acid dyes may be preferred on account of their ease of dyeing, provided that they possess the necessary fastness properties. Dyes in this class are sodium salts of organic sulphonic or carboxylic acids which, when dissolved in water, give an organic ion capable of being adsorbed by wool. The dye anions are of relatively low molecular weight and in general rely for their colour on the presence of azo groups or anthraquinone nuclei, the former giving rise to the yellow and the red dyes and the latter the blues and the violets.

Unfortunately the situation can arise where a dye satisfactory in all major properties is deficient in some minor respect. An example of this is the susceptibility of certain of these dyes to brine. When clothing or household furnishings have sea-water spilt on them, as could well happen in a seaside town, it is found with certain dyes that, when the salt water has evaporated, the material is discoloured in the area which has been wet. The effect is most noticeable when the material has been dyed with mixtures to give browns, greys, etc., and is in general shown by a yellowing of the colour. With single dyes brine spotting leads to a reduction in depth of colour. These changes are reversible, since the colour can be brought back to the original by treatment of the material with a warm dilute solution of acetic acid, and therefore they do not arise from destruction of dye. Colour changes such as these are mainly confined to certain of the blue level-dyeing acid dyes and may be a deterrent to their use.

Other examples of colour changes which can occur are the reddening of dyeings of Lissamine Ultra Sky R on treatment with fairly strong acid, and the loss of depth of dyeings of Solway Blue A when treated with alkali.

At the outset it seemed to the authors that these changes must be associated with the physical chemistry of these compounds. In other words, the changes which result from the treatment of dyed fibres with salt, acid, or alkali must be paralleled by changes which occur on addition of these reagents to solutions of the dyes. The work presented here makes such a comparison, and attempts to interpret the results in terms of present

knowledge of the colour of anthraquinone compounds in relation to their constitution.

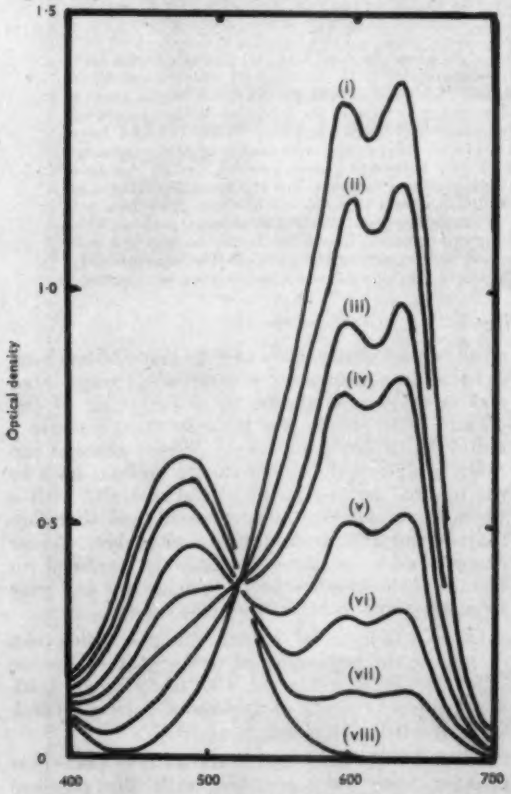
Reactions in Solution

Three aspects were examined—the colour changes which occur on the addition of (1) acid, (2) alkali, and (3) salt to aqueous solutions of the dyes. Eight examples of the level-dyeing acid blues were studied. Solutions of these dyes were made up in water to such a strength as to give a suitable optical density for spectrophotometric measurement. The dyes used were commercial samples which had been purified by precipitation and recrystallisation as the free acid where such a procedure was possible. One dye, Solway Ultra Blue B, proved intractable to such a treatment and was used as the commercial sodium salt. Comparative experiments using the commercial sodium salts and the purified free acids of several of the dyes showed no significant difference in the various quantities measured. Additions of acid, alkali, or salt were made to these solutions, and the absorption spectra measured by means of a Cary recording spectrophotometer. The results are discussed separately.

1. ADDITIONS OF ACID

Since the dyes are blue in colour, they are characterised by an absorption band in the 575–675 mμ. region of the spectrum, this band being for many of them a "double-headed" peak. In general, the changes on addition of acid are very marked with all the dyes. There is very little change in spectrum at the low concentrations of acid, but with increasing concentration a point is reached at which the intensity of absorption falls rapidly. This is accompanied by the development of a band at shorter wavelengths, whose position depends on the particular dye being studied.

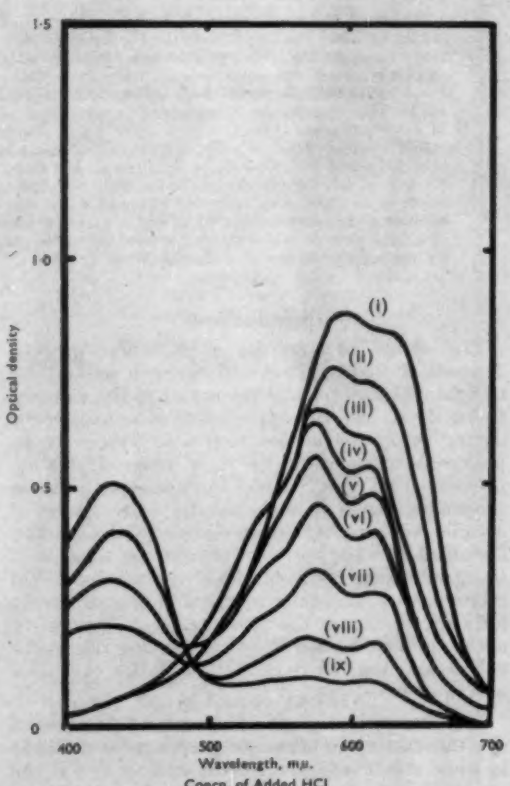
An example is shown in Fig. 1, which gives the spectra of Lissamine Ultra Sky R solutions containing increasing amounts of hydrochloric acid. The spectrum in the absence of acid shows a double-headed peak, which decreases in intensity as the acid concentration increases. This change is accompanied by the development of a peak at 476 mμ.,. giving an orange–yellow coloured solution. The spectra in Fig. 1 are indicative of a system confaining two and only two species, all curves passing through a common point of intersection, an *isosbestic* point.



Concn. of Added HCl

(i)	0	(v)	0·0147 N.
(ii)	0·00117 N.	(vi)	0·0343 N.
(iii)	0·00351 N.	(vii)	0·0986 N.
(iv)	0·00585 N.	(viii)	2·350 N.

FIG. 1— Effect of Acid on Absorption Curve of Aqueous Solutions of Lissamine Ultra Sky R (Free Acid)



Concn. of Added HCl

(i)	0	(vi)	4·60 N.
(ii)	0·013 N.	(vii)	5·76 N.
(iii)	0·133 N.	(viii)	6·91 N.
(iv)	0·921 N.	(ix)	8·06 N.
(v)	3·45 N.		

FIG. 2— Effect of Acid on Absorption Curve of Aqueous Solutions of Solway Blue BN (Free Acid)

Some of these dyes show more complicated behaviour, e.g. Solway Blue B (Fig. 2). This dye, too, has a double-headed peak, at 595 mμ. and 625 mμ. The optical density of the longer-wavelength band decreases as the acid concentration increases, but initially there is a smaller decrease in the shorter-wavelength band. The explanation of this behaviour lies in the fact that the colour changes occur in two steps: the first of these is shown by the development of a "hump" at approx. 535 mμ., which is undoubtedly due to the first product of the reaction of acid with the dye. The reduced effect on the optical density of the peak at 595 mμ. is due to the development of the spectrum of this compound. With further increase in acid concentration the system behaves normally, in that the intensity of both peaks decreases rapidly with the simultaneous development of a new peak at much shorter wavelengths (437 mμ.). The spectra do not pass through a well defined isosbestic point, in agreement with the presence of more than two species.

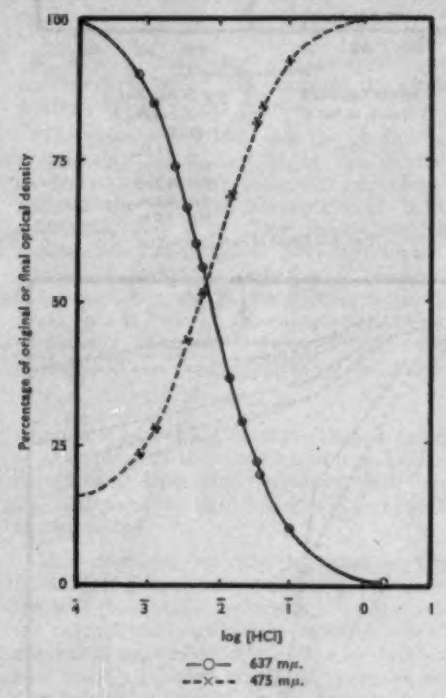


FIG. 3.—Titration Curve of Lissamine Ultra Sky R (Free Acid) in Water

For quantitative assessment of the differences between dyes, the changes described above are shown more clearly when the percentage reduction or increase in optical density of the peaks is plotted against the logarithm of the amount of acid added to the dye solution. For those dyes which have only one stage in the reaction, e.g. Lissamine Ultra Sky R, this kind of plot gives a result typical of an acid–base titration (Fig. 3). With a dye which changes its spectrum in two stages (such as Solway Blue B), the changes in the optical density of the long-wave peak show two steps, a smaller one (*A* in Fig. 4) followed by the main one (*B*) at higher concentrations of acid.

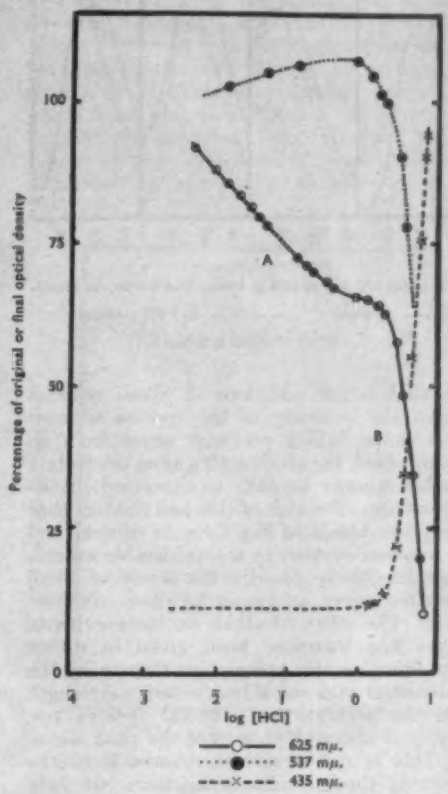


FIG. 4.—Titration Curve of Solway Blue BN (Free Acid) in Water

In Fig. 4 is also plotted the development of the band of shortest wavelength together with the changes at the wavelength of the hump (537 mμ.). At this last point in the spectrum, the optical density first increases as acid is added, and then falls rapidly as the density at the main peak falls. This is to be expected, as the system reacts with acid in two stages. Of the dyes studied only Solway Blues A, B, and G showed two-stage reactions.

The differences between the dyes have been described in terms of dissociation constants derived from the data. The calculation of these is described later.

2. ADDITIONS OF ALKALI

Similar experiments have been carried out in which sodium hydroxide was added to the dye solutions in place of hydrochloric acid. For five of the dyes there was no change in the shape of the spectral curve (e.g. Solway Blue R); for one dye (Solway Blue G) the changes were very small; whilst in the extreme case (Solway Blue A) the original spectrum was changed to give a new peak at a longer wavelength. The effects are shown in Fig. 5. The situation is, however, complicated by

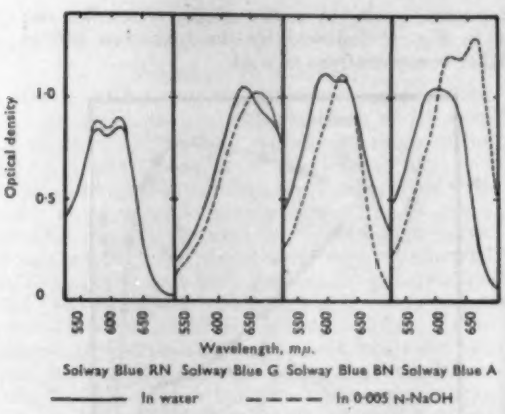

Optical density
1·0
0·5
0
550 600 650 550 600 650 550 600 650 550 600 650
Wavelength, mμ.
Solway Blue RN Solway Blue G Solway Blue BN Solway Blue A
In water
In 0·005 N-NaOH

FIG. 5— Effect of Alkali in Solution

the fact that larger additions of alkali cause a decrease in the intensity of the spectra without change of shape, which probably arises from an aggregating effect, the alkali acting as an electrolyte in a similar manner to salt, as described in the following section. Because of this and the fact that the absorption bands of the dyes in neutral and alkaline solution overlap to a considerable extent, it was not feasible to describe the action of alkali by a titration curve analogous to those obtained using acid. The effect of alkali on the spectra of these dyes has therefore been given in rather arbitrary units as the percentage change in the optical densities at a suitably selected wavelength for both the alkaline and neutral spectra respectively, and also as a change of the peak wavelength. This is not a serious drawback to the assessment of these results, since there are only three dyes which show marked changes (Solway Blues A, B, and G), and it is clear from the spectra shown in Fig. 5 that the Blue A changes to a considerably greater extent than the Blue B, and that both change more than Solway Blue G, the only other dye which is affected.

3. ADDITIONS OF SALT

Addition of salt to solutions of these compounds in water causes loss in depth, as may be seen from the examples given in Fig. 6. It is clear that there is neither a change in shape of the curve nor a simultaneous development of an absorption band in another part of the spectrum, the optical density of the solution merely decreasing until the dye precipitates. As a precautionary measure these solutions were measured after standing for 18 hr., to ensure that any changes arising from the salt additions were complete. The data have been plotted in a similar fashion to those for the previous experiments, i.e. percentage change in optical density against the concentration of salt in solution (Fig. 7). The graphs show clearly that the additions of salt in several cases cause continuous decreases in optical density until obvious precipitation is observed. It is interesting at this juncture to note that the concentrations of salt required to show substantial changes in optical density are considerably higher than either the acid or the alkali concentrations necessary in the previous experiments.

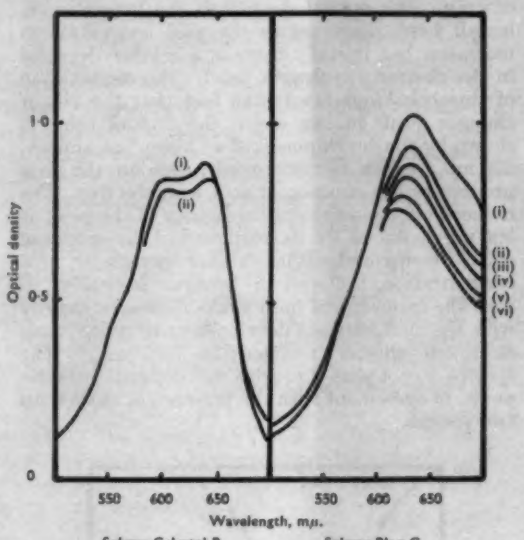


FIG. 6— Effect of Salt in Solution

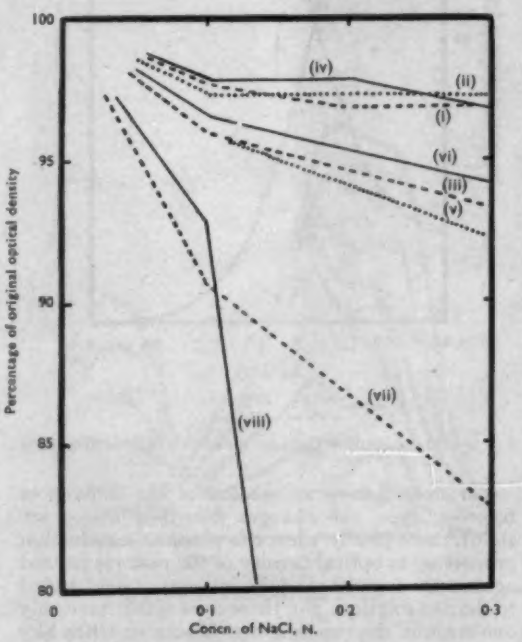


FIG. 7— Effect of Sodium Chloride in Solution

Colour and Constitution of Anthraquinone Derivatives

Any explanation of the spectral changes described above must be associated with the relationship between the position of the absorption bands and the chemical constitution of the dyes. For this reason, before attempting to associate these changes with results of tests on dyed wool, it seems worthwhile to sketch some of the salient points of present knowledge of the colour of anthraquinone compounds in relation to their chemical constitution.

Two concepts have formed the basis of most modern theories of the origin of colour in organic molecules. Firstly, the molecule must possess a group which is a potential colour-forming system. These are generally unsaturated groups of the type

$$-N{:}O \quad \rangle C{:}C\langle \quad -N{:}N- \quad \rangle C{:}O$$

and are called chromophores. Secondly, another set of groups is required, the auxochromes, whose presence is necessary to develop the latent colour of the chromophore. By contrast these are saturated, salt-forming groups of the type –OH, $-NH_2$, –NHR, and $-NR_2$. It is the interpretation of the above effects which has occupied the attention of workers during recent years [1].

The years about 1920 saw the beginning of the present-day belief that it is the oscillation of electrons within the molecular structure which permits the selective absorption of light by a substance, and it is along these lines that the modern theories of colour have developed. When visible or ultraviolet radiation is absorbed by a compound, the energy transferred from the light beam goes to energise the electrons in the molecule, the energy absorbed E being related to the frequency ν of the absorbed light by the expression—

$$E = h\nu$$

where h is Planck's constant. Thus if the energies of absorption of the electrons in a molecule can be determined, then the frequency and hence the wavelength of the light which will be absorbed can be calculated.

This approach may be regarded as the most fundamental and has been applied very satisfactorily to simple molecules, but unfortunately the calculation becomes extremely involved when applied to molecules as complex as those of dyes, and hence a simpler, empirical approach has been adopted. Briefly the basic principles may be described as follows.

In any compound possessing a system of alternate double and single bonds, i.e. a conjugated system, the electronic configuration may be written down in a number of ways, for example I—

$$\underset{(a)}{C{=}C{-}C{=}C} \qquad \underset{(b)}{\overset{+}{C}{-}C{=}C{-}\overset{-}{C}} \qquad \underset{(c)}{\overset{-}{C}{-}C{=}C{-}\overset{+}{C}}$$

(I)

The forms (*b*) and (*c*) are the extremes to which the system tends and which result from the oscillation of electrons within the molecule. Classical mechanics predicts that the wavelength of such a linear oscillator is determined by its length, and this is in fact found to be so, the wavelength of maximum absorption of compounds containing such conjugate chains increasing with the length of the chain. The function of the auxochrome is to enhance this possibility of electron oscillation, and any group which is capable of increasing the contribution of the optically important ionic forms will develop the colour of the substance. These ideas may be illustrated by application to substituted anthraquinones.

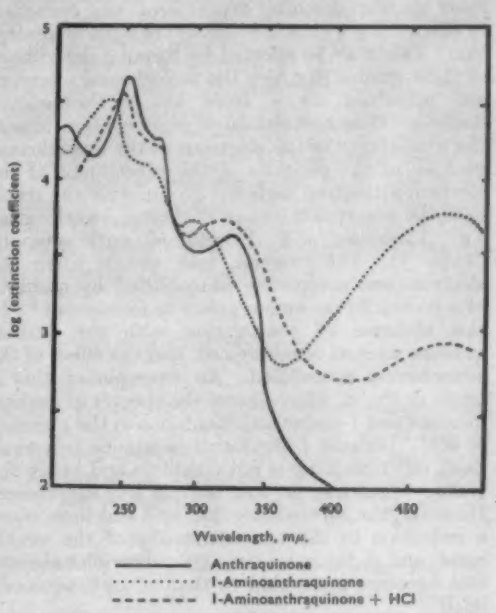


FIG. 8— Solutions in Methanol

Anthraquinone itself is practically colourless, all the absorption bands of any importance occurring in the ultraviolet region of the spectrum (Fig. 8). This curve may be regarded as characteristic of the anthraquinone chromophore, and will be modified by the introduction of an auxochrome. Thus the presence of an amino group produces a very strong band in the visible region, with only a comparatively small change in the ultraviolet spectrum.

The action of the auxochrome in developing the colour may be interpreted as arising from interaction of its electrons with those of the chromophore. In the case quoted, since the amino group has a lone pair of electrons, the interaction will be strong, and in general a powerful auxochrome will be a group whose electrons may readily interact. Auxochromes thus act to different degrees depending on their electronic properties, and it is possible to write them down in series in order of their effects. Thus, if the various groups are substituted successively in anthraquinone, the wavelength of maximum absorption increases in the order: $H < OH < NH_2 < NH{\cdot}CH_3 < O^- < N(CH_3)_2$ (Table I). This order has been

TABLE I

Wavelengths (mμ.) of Maximum Absorption of Substituted Anthraquinones in Methanol Solution

H	323	1-OH ...	402	1-NH_2 ...	475
1-O^-	493	1:5-$(OH)_2$ ...	417	1:5-$(NH_2)_2$ ...	487
1-NH CH_3 ...	503	1:8-$(OH)_2$ ...	428	1:8-$(NH_2)_2$ ...	500
1-$N(CH_3)_2$ ...	503	1:4-$(OH)_2$ ...	483	1:4-$(NH_2)_2$ ...	550–590
1-NH-CO-CH_3	400	1:4:5-$(OH)_3$	492	1:4:5-$(NH_2)_3$	561–601
1-NH-CO-C_6H_5	405	1:4:5:8-$(OH)_4$	552	1:4:5:8-$(NH_2)_4$	600–629

demonstrated in other series of compounds, such as acetophenones, benzoic acids, and nitrobenzenes.

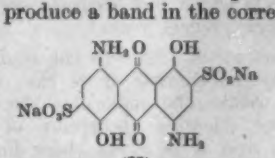
The substituents given above fall in the order of their electron-donating capabilities; any reduction in this ability will reduce the wavelength of absorption This may be effected by forming derivatives of these groups in which the auxochrome electrons are attracted away from the anthraquinone nucleus. Thus acetylation or benzoylation lessens the availability of the electrons in the auxochrome because of the presence in the substituent of the electron-attracting carbonyl group, with the result that the absorption occurs at shorter wavelengths, e.g. 1-amino- and 1-acetamido-anthraquinone (Table I). The extreme case occurs when the electrons are completely immobilised by addition of a proton to the amino group to form a salt [2]. In this instance all conjugation with the anthraquinone nucleus is suppressed, and the effect of the auxochrome is nullified. An example of this is given in Fig. 8, which shows the spectra of anthraquinone and 1-aminoanthraquinone in the presence of acid. Because 1-aminoanthraquinone is a weak base, salt formation is not complete and hence the visible spectrum is not completely suppressed. However, the curves show that acid additions cause a reduction in the optical density of the visible band, and at the same time the ultraviolet absorption becomes more akin to that of anthraquinone itself.

There are other ways in which the spectrum of a compound may be modified, e.g. by aggregation. It has been demonstrated with the cyanine dyes that it is possible to form a completely new absorption band by increasing the concentration of the dye in solution [3]. In other instances a dye solution may show a shoulder on the side of the main absorption band. Increasing the concentration causes this shoulder to grow at the expense of the main peak until the situation is reversed. An example of this behaviour occurs with Methylene Blue [4]. With other dyes, however, aggregation may be shown only by small changes in the intensity of absorption. In all instances these phenomena are reversible, the spectra returning to those in dilute solution when the more concentrated solutions are diluted.

APPLICATION TO ANTHRAQUINONE BLUE DYES

All the compounds in this class contain two or more substituents capable of acting as auxochromes. The peak wavelengths of such compounds depend to a marked extent on the position as well as the nature of the substituents. Thus the 1:5- and 1:8-disubstituted derivatives show single peaks at wavelengths quite close to those of the corresponding monosubstituted compounds, whereas the 1:4-disubstituted anthraquinones show marked differences and often possess double-headed peaks (Table I).

To obtain a blue compound from anthraquinone requires at least two and frequently three auxochromes, each capable of supplying electrons to the nucleus (e.g. groups of the amino and hydroxyl types). Thus in the series: 1-amino-, 1:4-diamino-, 1:4:5-triamino-, and 1:4:5:8-tetra-amino-anthraquinone the bands have maxima at 475, 550–590, 561–601, and 600–629 mμ., but it is not until the triamino derivative is reached that the compound could be described as blue. A typical structure of an acid blue dye is that of Solway Blue B (II, *C.I.* 1054), which has four of these powerful auxochromes to produce a band in the correct position.



(II)

The action of acid on these compounds can readily be explained in terms of salt formation, i.e. the formation of R·NH_3^+ groups. This immobilises the lone pair of electrons on the amino group and hence nullifies its auxochromic properties. The band which develops on addition of acid is therefore due to the compound without the amino group. For example, the addition of acid to 1-amino-4-hydroxyanthraquinone reduces the intensity of the absorption band and simultaneously increases that of a band corresponding to 1-hydroxyanthraquinone.

The titration curves described earlier for the level-dyeing blue dyes can therefore be regarded as measuring the addition of a hydrogen ion to the amino groups in the dyes and can be used to calculate the equilibrium constant K_H of the reaction—

$$D\cdot NH_3^+ + H_2O \rightleftharpoons D\cdot NH_2 + H_3O^+$$

since—

$$K_H = \frac{[D\cdot NH_2]\,[H_3O^+]}{[D\cdot NH_3^+]}$$

From K_H the basic dissociation constant of the amine K_b is easily obtained from—

$$K_b = \frac{K_w}{K_H}$$

where $K_w = 10^{-14}$ for water. For the present purpose, K_H is the more useful figure, since it gives a direct measure of the concentration of acid required to reduce the amount of amine to 50% of

its initial value. Actually, pK_H ($= -\log K_H$) is still more convenient for representing relatively small values of K_H. The constants obtained inthis way are listed in Table II, each result being the average of at least two experiments. The variation between repeats was of the order of 1–5% (K_H), depending on the shape of the titration curve, an accuracy which was considered adequate for the purposes of the correlations made in this paper.

TABLE II

Dye	pK_H	K_b
Lissamine Ultra Sky R	2·09	$1·2 \times 10^{-13}$
Solway Celestol B ...	1·22	$1·7 \times 10^{-13}$
Solway Ultra Blue B ...	0·92	$8·3 \times 10^{-14}$
Solway Blue RN ...	0·74	$5·7 \times 10^{-14}$
Solway Blue A ...	0·64	$4·5 \times 10^{-14}$
	(−1·36*)	($4·4 \times 10^{-13}$*)
Solway Blue BN ...	0·60	$4·0 \times 10^{-14}$
	(−1·43*)	($3·7 \times 10^{-13}$*)
Solway Blue G ...	0·44	$2·8 \times 10^{-14}$
	(−1·53*)	($3·0 \times 10^{-13}$*)
Solway Blue 2G ...	0·01	1×10^{-14}

* Second dissociation

It is of interest to note that, in dyes which contain more than one group, the titration goes in two stages (e.g. Solway Blue BN), the absorption of the intermediate compound merely showing as a hump on the side of the spectral curve. This hump will be due to a spectrum similar to that of 1-amino-4:8-dihydroxyanthraquinone-3:7-disulphonic acid. At the higher acid concentrations, the spectrum becomes similar to that of 1:5-dihydroxyanthraquinone-2:6-disulphonic acid.

The spectral changes which occur on the addition of alkali are in agreement with the rules sketched above. If the dye contains a hydroxyl group, then alkali is able to transform this to the more powerful O^- group, with a corresponding shift of the spectrum to longer wavelengths, as is seen from the wavelengths of such compounds in Table I.

The explanation of the behaviour of the dye solutions on addition of salt is not so clear-cut, although it seems likely that reduction in intensity of the absorption is due to aggregation of the dye. Such a suggestion is not incompatible with curves of the type given in Fig. 6, which could represent an aggregation process, followed finally by salting out of the dye. Unfortunately there is no direct evidence for this hypothesis, and the interpretation in terms of aggregation is made both by analogy with other systems, and also because the presence of an organic solvent (e.g. methanol) results in a marked reduction of the effect. In this latter case it is usually considered that the presence of a relatively hydrophobic solvent helps to disperse the dye.

The Behaviour of the Dyes on Wool

The major practical interest in studying the properties of these dyes in solution lies in whether such a study will provide an explanation of the colour changes which occur when dyed patterns are treated with these reagents. Samples of wool dyed with these dyes have been examined, and the results assessed in the light of the experiments previously described.

1. TREATMENT WITH ACID

Pieces (each 5 g.) of botany serge were dyed with each dye to a convenient depth (ca. 0·2%) for optical measurements. Circular samples about $1\frac{1}{4}$ in. in diameter were cut from the piece and shaken at room temperature for 30 min. in 50 ml. of 0·1 N., 0·2 N., and 0·5 N. solutions of sulphuric acid. The samples were then removed from the acid, squeezed in the hand, and allowed to dry in the air in a Petri dish. The reflection spectra of the samples were measured before and after treatment by means of the General Electric recording spectrophotometer (a single thickness was used, as preliminary experiments had shown that this was adequate to give an infinitely thick layer, i.e. one for which further increase in thickness gave no significant change in reflection). The colour changes are described in terms of the change in reflectance at the wavelengths of the absorption band of the dye. In Table III the effects caused by 0·5 N. sulphuric acid are given. The figures are expressed in terms of the equivalent concentration of dye; i.e., since the reciprocal of the reflectance value has been shown to be proportional to the dye concentration [5], the expression—

$$\frac{\frac{1}{R_1} - \frac{1}{R_2}}{\frac{1}{R_1} - \frac{1}{R_0}}$$

(where R_1 and R_2 are the reflectances of the untreated and treated patterns and R_0 is that of the undyed wool) gives a value which is equivalent to the decrease in the effective concentration of dye due to the treatment.

TABLE III

Effect of 0·5 N. Sulphuric Acid on Dyeings on Wool

Dye	pK_H	Loss in Depth (%)	Fastness to Carbonising
Lissamine Ultra Sky R	2·09	60	2
Solway Celestol B ...	1·22	27	4–5
Solway Ultra Blue B ...	0·92	7	4–5
Solway Blue RN ...	0·74	14	5
Solway Blue A ...	0·64	20	4
Solway Blue BN ...	0·60	14	4
Solway Blue G ...	0·44	0	4
Solway Blue 2G ...	0·01	6	4

In the second column of Table III are given the dissociation constants of the dyes as deduced from the titration curves described earlier. A comparison of the two columns shows that the maximum colour change occurs with Lissamine Ultra Sky R, the dye with the highest value for pK_H and hence the most sensitive to acid. The other dyes, with smaller values of pK_H, show smaller effects.

In the last column of the table are quoted the fastness-to-carbonising figures given in the I.C.I. pattern card. It can be seen that there is only one dye which is poor in this respect, and that is again Lissamine Ultra Sky R, with the largest value of pK_H.

2. TREATMENT WITH ALKALI

In investigating this property an attempt was made to estimate the colour changes which occur when dyed wool is treated with sodium carbonate. This was done by wetting the patterns in sodium carbonate solution (50 g./litre) for 30 sec., squeezing, repeating the treatment, and then drying in air. The percentage changes were assessed as above. The changes in solution (Fig. 5) are simply described by the percentage difference in optical density, at the selected wavelength, between the neutral and the 0·005 N. caustic soda solutions. These figures, together with the wavelength shift which occurs on the addition of alkali, are compared in Table IV with the fastness data.

was then allowed to dry slowly in a desiccator. Reflection spectra were recorded before and after this treatment, and the changes noted. For five of the eight dyes the treatment merely resulted in a uniform increase in the reflection at all wavelengths, but for the others the level of reflection was not raised uniformly, the change being greater at the peak absorption of the dye.

The results are not so easy to interpret, since significant changes in spectrum are masked to a considerable extent by the deposition of salt in the interstices of the material, giving rise to a general raising of the reflection. In Fig. 9 some spectra are given which demonstrate this effect.

TABLE IV

Change in Colour on Treatment with Alkali

Dye	0·005 N-NaOH in Soln.		Na_2CO_3 (50 g./litre) on Wool Patterns		Fastness to Alkali (visual assessment)
	Change in Optical Density at Selected λ (%)	Change in Peak λ (mμ.)	Change in Depth at Selected λ (%)	Change in Peak λ (mμ.)	
Solway Blue A ...	230	63	40	ca. 50	1–2
Solway Blue BN ...	29	25	21	4	2
Solway Blue G ...	14	21	27	4	3–4
Solway Celestol B ...	Little or no change		—	—	3–4
Solway Blue RN ...			No change	No change	4
Solway Ultra Blue B ...			—	—	4
Solway Blue 2G ...			—	—	4
Lissamine Ultra Sky R...			—	—	4

It is seen that dyes with a low rating for fastness to alkali show large changes in solution.

An interesting point about these colour changes may be mentioned: addition of alkali to a solution of Solway Blue G (Fig. 5) results in a change in spectrum of a similar magnitude to that obtained with Solway Blue BN; visually, however, the change is less.

This apparent paradox is explained by the fact that the response curve of the eye is falling off rapidly as the wavelength becomes longer, being almost zero at 700 mμ., and Solway Blue G absorbs nearer to this critical wavelength (635–675 mμ.) than Solway Blue BN (595·4–625 mμ.). Modifications in the spectrum of the former will not be as apparent to the eye.

3. SALT SPOTTING

The treatment of dyed materials with salt can on occasions lead to changes in hue and depth. To measure the effect of salt on the dyes under consideration, botany serge was dyed with the various dyes to about 0·2% depth, both singly and in combination with Lissamine Yellow 2G and Azogeranine 2G to give brown dyeings of suitable depth. The patterns were dyed in the usual way, rinsed in cold water, and then dried in air. From these were cut circles of about 1½ in. diameter, which were placed in Petri dishes of a suitable size. The samples were treated with sufficient brine solution (2 c.c. of a solution containing 30 g. sodium chloride, 6 g. calcium chloride, and 1 c.c. Lissapol N per litre) just to wet the piece, which

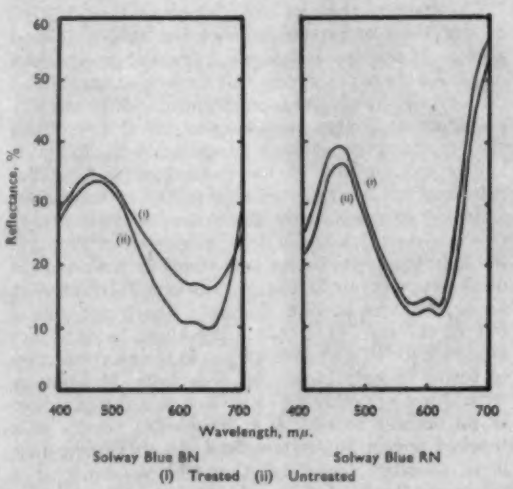


FIG. 9—Reflectance of Dyed Patterns before and after Treatment with Brine

The changes which occur on allowing salt solution to evaporate on the material undoubtedly arise from the high concentrations which are developed. Two causes for the sensitivity of these dyes to salt could be put forward: either the salt liberates free mineral acid from the fibre, which is then able to react with the dye to form the amine salt as shown earlier; or the dye is detached from the fibre by the competition of the chloride ions

for the amino group sites, and, once released, is capable of aggregating with consequent reduction in depth of colour, as demonstrated by the experiments in which salt was added to solutions of the dyes in water.

The liberation of acid during the drying in presence of salt was demonstrated by treating material dyed with a suitable indicator in a similar manner. The indicator chosen was Metanil Yellow YK, which changes from yellow to violet at pH values between 1·2 and 2·3. This indicator, when applied from the usual acid dyebath, dyed wool yellow, and showed marked changes in hue towards red when the material was dried in the presence of salt, thereby indicating a release of acid from the fibre. These changes did not occur when the Metanil Yellow had been applied from a neutral dyebath, salt having no effect on the resulting dyeings. Thus it seemed at first sight that the changes caused by salt spotting should parallel the values of K_H previously determined.

Table V shows the comparison between the K_H values and the results on wool, given as the average of the results of experiments on the two sets of patterns, and expressed as described previously by the percentage change at the wavelength of maximum absorption. There appears to be no relation between the two properties.

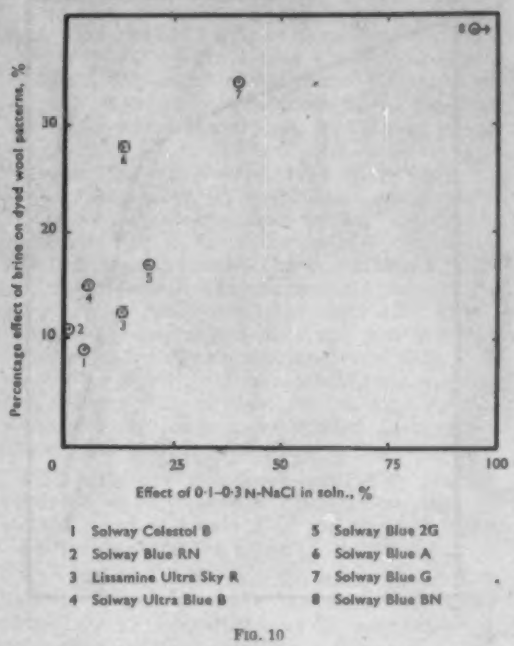


FIG. 10

TABLE V

Effect of Brine on Colour

Dye	Dyeings on Wool: Fastness Assessment	Dyeings on Wool: Decrease in Optical Density (%)	pK_H	Dye Soln. (0·1–0·3 N-NaCl)	Affinity of Dye for Wool (kcal./mole)
Solway Blue BN ...	2	39	0·60	−470 (ppt.)	4·49
Solway Blue G ...	3	34	0·44	− 40	4·35
Solway Blue A ...	3	28	0·64	− 13	4·12
Solway Blue 2G ...	3–4	17	0·01	− 19	—
Solway Ultra Blue B ...	4	15	0·92	− 5	5·90
Lissamine Ultra Sky R	4–5	13	2·09	− 13	5·28
Solway Blue RN ...	4	11	0·74	0	5·22
Solway Celestol B ...	3–4	9	1·22	− 4	5·29

In the fifth column of Table V are given the changes in optical density produced in a dye solution by increasing the concentration of salt from 0·1 N. to 0·3 N., and these may be compared with the figures for the changes on wool. The agreement here is very much better, and may be demonstrated in Fig. 10, where the two properties are plotted one against the other, there being only one exception in Solway Blue A. It is significant, too, considering the ease with which Solway Blue BN precipitates, that small particles of wool dyes have been found by Astbury [6] and Royer [7] in wool fibres by microscopic examination of cross-sections.

However, before either of these effects can be fully assessed, it must be realised that combination of the dye with the fibre will modify the properties of the dye which are discussed here. Thus the effect of titrating solutions of the dye in the presence of gelatin is to displace the titration curve to lower pH values (Fig. 11); i.e. more acid is required to effect any particular change. It seems likely, therefore, that for acid attack on the dye to occur, the dye will have to be desorbed from the fibre. Similarly, if the dye molecules are to become capable of migrating and aggregating under the influence of salt, then they must first be desorbed. With these facts in mind, it was thought necessary to measure the ease with which these dyes may be removed from the fibre, i.e. to measure their affinities. These were determined by a desorption technique described elsewhere [8]. The last column of Table V shows that those dyes which have low affinities are the most susceptible to salt. Thus, Solway Blue A will be desorbed to a much greater extent than Lissamine Ultra Sky R, and hence will be able to be more affected by salt. It appears, therefore, that the ability to be desorbed is of prime importance in subsequent attack by acid or aggregation by salt.

From the point of view of deciding between the two latter effects, it is unfortunate that reflectance spectra do not give much help in differentiating

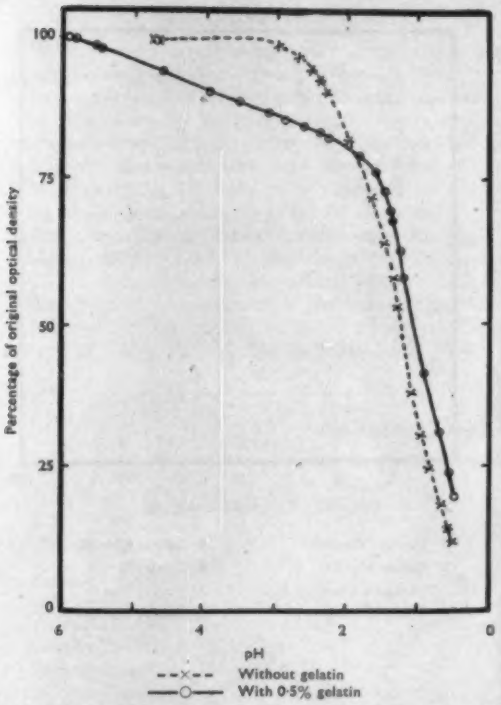


FIG. 11— Effect of Gelatin on Sensitivity to Acid of Solway Celesto B in Aqueous Solution

between them, i.e. indicating whether or not a colour change occurs on salt-spotting. This difficulty is enhanced by the fact that the colour change involved in the first stage of acid attack (e.g. with Solway Blue B) is only small for those dyes which are affected by salt.

The evidence described above shows that the changes brought about by salt-spotting run parallel with the affinities of the dyes and with their sensitivities to salt in solution, but there is some doubt whether part of the change can be associated with mineral acid liberated from the fibre.

DYEHOUSE LABORATORIES
IMPERIAL CHEMICAL INDUSTRIES LTD.
DYESTUFFS DIVISION
HEXAGON HOUSE
MANCHESTER 9

(*MS. received 8th September 1955*)

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The Spectral Regions of Daylight which cause Fading

K. MCLAREN

Organic colouring matters are faded by absorbed ultraviolet and visible radiation up to a critical wavelength, above which radiation is non-actinic even if strongly absorbed. This wavelength is generally related to the normal light fastness of the colouring matter, "normal" referring to the substrate for which it was originally developed, e.g. vat dyes on cotton, disperse dyes on acetate rayon. The critical wavelength decreases as the resistance to fading increases, being in the red when the light fastness is 1–2 (minimum) and in the blue when it is 6–8 (maximum); there are, however, some exceptions to this rule.

As long-wave radiations preponderate in daylight and as the absorption bands are usually strongest in the visible spectrum, fugitive organic colouring matters are mainly faded by visible light, whilst those of high normal light fastness can in general be faded only by ultraviolet, violet, and blue light.

The differences between the composition of actinic radiation constituting daylight and that emitted by the enclosed carbon arc have been shown to be relatively unimportant as a possible source of *gross* anomalies in light fastness testing based on comparison with light fastness standards. These differences do, however, render meaningless any attempts to equate one hour's lamp exposure with so many hours' sunshine.

Introduction

It is well known that light fastness assessments made by exposure in fading lamps often give results significantly different from those made by exposure to daylight, and an investigation into the causes of these discrepancies is at present being made by the Light Fastness Subcommittee. It has often been suggested that the excessive ultraviolet radiation emitted by the enclosed carbon arc is responsible, although this has never been proved. This possibility was considered in detail[1], but a really satisfactory conclusion could not be reached, as it was not known which photochemically active radiations (295–800 mμ.) present in daylight were responsible for the fading of coloured materials. The Grotthus–Draper law* implied, for example, that when a bright greenish-yellow pattern fades, violet light and/or ultraviolet radiation must be responsible, as the pattern does not absorb any other photochemically active radiation: the con-

*"Only radiations which are absorbed by the reacting system are effective in producing chemical change."

verse, however, could not be assumed; e.g. green patterns absorb both red and blue light, but are not necessarily faded by either or both.

To enable the importance of spectral composition to be put in its proper perspective, therefore, an investigation was undertaken into the regions of daylight which cause fading.

Experimental

Although fundamental investigations into photochemistry are ideally undertaken with monochromatic radiations of known and constant intensity, such refinement is inevitably arduous, and in this investigation was considered quite unnecessary for the strictly practical information required. A much simpler method was therefore devised, which enabled adequate information to be obtained in a very short time with little effort.

The method consisted in exposing the material under examination behind a set of five filters, a refinement of the method previously employed by Appel and Smith[2]. Plastics filters were used instead of glass in this investigation, however, because of their higher transmission and steeper cut-off. The five filters used are listed in Table I, and their transmission curves are shown in Fig. 1.

Table I

Filter	Absorption Characteristics
(i) Perspex Red 400 (⅛ in. thick)	Absorbs > 99·9% of radiations < 600 mμ.
(ii) Perspex Yellow 200* (⅛ in. thick)	Absorbs > 99·9% of radiations < 460 mμ.
(iii) Cellulose acetate containing Uvinul 490 (GDC)	Absorbs > 99·9% of radiations < 400 mμ.
(iv) Perspex containing another ultraviolet absorber	Absorbs > 99·9% of radiations < 360 mμ.
(v) Perspex Clear	Transmits all actinic radiation present in sunlight

* Perspex Yellow 200 has a transmission band in the ultraviolet at 335 mμ. To remove this, a piece of the ultraviolet-absorbing Perspex was superimposed on the yellow.

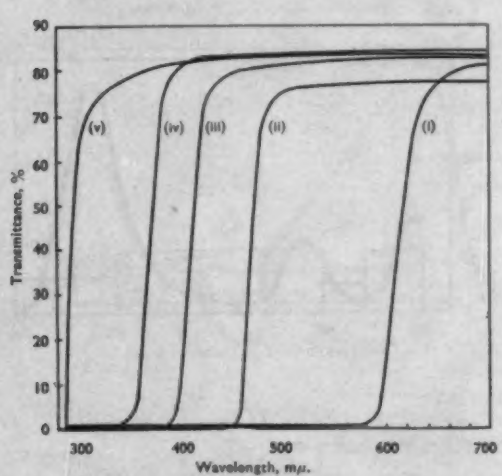

Fig. 1—Transmission Curves of the Filters used in the Fading Experiments

The filters were mounted side by side, and in use a set incorporating a strip of aluminium to provide an unexposed reference area was placed over a sample of the coloured material of the same size (5 in. × 2 in.) as the composite filter. The filter assemblies were then exposed in the open facing due south at 45° to the horizontal on sunny days between May and October 1954 in Cheshire, and between November 1954 and June 1955 in Bombay. Owing to the poor summer in England, it was inevitable that in some cases part of the former exposure was made under cloudy conditions, which probably means that too much reliance should not be placed on the quantitative data obtained for the 295–360 mμ. region; this was not, however, regarded as a serious limitation of the method, in view of the purely practical aim of the investigation and the fact that all exposures for light fastness assessment are made under window glass, which absorbs much of this radiation.

Exposure was continued until the contrast produced under the clear Perspex was approximately equal to Grade 2 of the Society's Grey Scale for assessing the Effect on the Pattern.

The reflectance curves of the unexposed portion and the five areas under the filters were then plotted on the General Electric Recording Spectrophotometer (Hardy), and the reflectances calculated at the point of maximum absorption.

Fig. 1 shows that the transmitting regions of each filter are approximately of equal value, viz. 80 ± 3%; it is therefore justifiable to calculate the amount of fading caused by each of the five different regions of the spectrum, and Tables II and III illustrate the method employed. By subtracting the increase in reflectance (Table II) caused by exposure under filter (i) from that under filter (ii), the effect of radiation of wavelengths between 460 and 600 mμ. can be calculated. The effects of other regions can be calculated similarly (Table III).

Table II

Chlorazol Azurine G on Paper

Filter	Radiation received (mμ.)	Reflectance at 620 mμ. (%)	Increase in % Reflectance on Exposure
Unexposed	—	11·5	—
(i)	> 600	12·6	1·1
(ii)	> 460	14·6	3·1
(iii)	> 400	15·8	4·3
(iv)	> 360	16·2	4·7
(v)	> 295	16·7	5·2

Table III

Spectral Region Wavelength (mμ.)	Colour	Increase in % Reflectance	Proportion of Total Effect (%)
>600	Infrared, red, orange	1·1	21
460–600	Yellow, green, blue	2·0	39
400–460	Violet	1·2	23
360–400	Near ultraviolet	0·4	8
295–360	Middle ultraviolet	0·5	9
			100

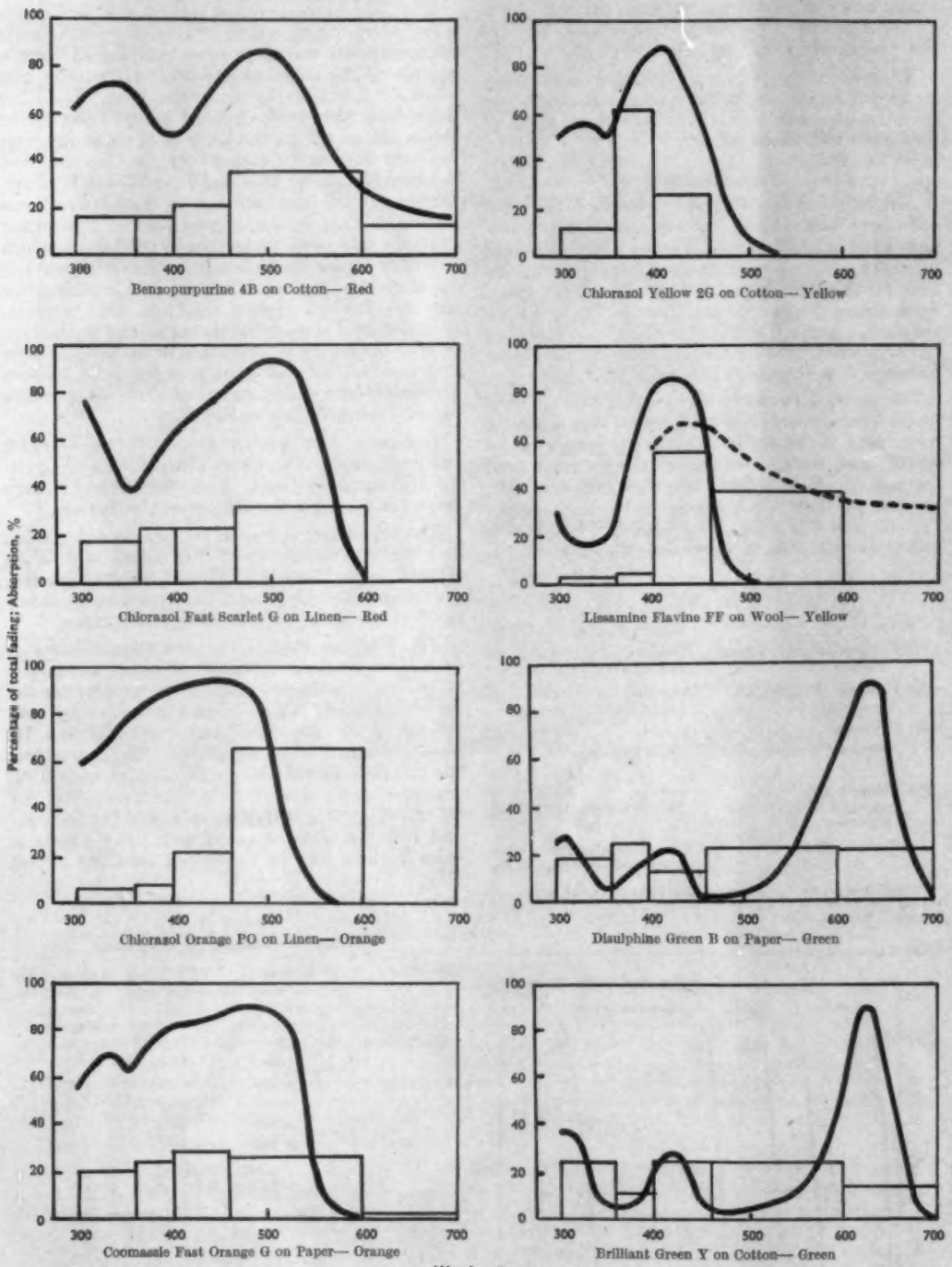
Percentage of total fading; Absorption, %
Benzopurpurine 4B on Cotton— Red
Chlorazol Yellow 2G on Cotton— Yellow
Chlorazol Fast Scarlet G on Linen— Red
Lissamine Flavine FF on Wool— Yellow
Chlorazol Orange PO on Linen— Orange
Disulphine Green B on Paper— Green
Coomassie Fast Orange G on Paper— Orange
Brilliant Green Y on Cotton— Green
Wavelength, mμ.
Absorption curve of dye solution
Absorption curve of dyed pattern
Proportion of total fading of dyed pattern due to given spectral region

FIG. 2— Diagrammatic Representation of the Contribution to Fading of Five Spectral Regions of Daylight together with the Absorption Spectrum of the Dye (Fugitive Dyes)

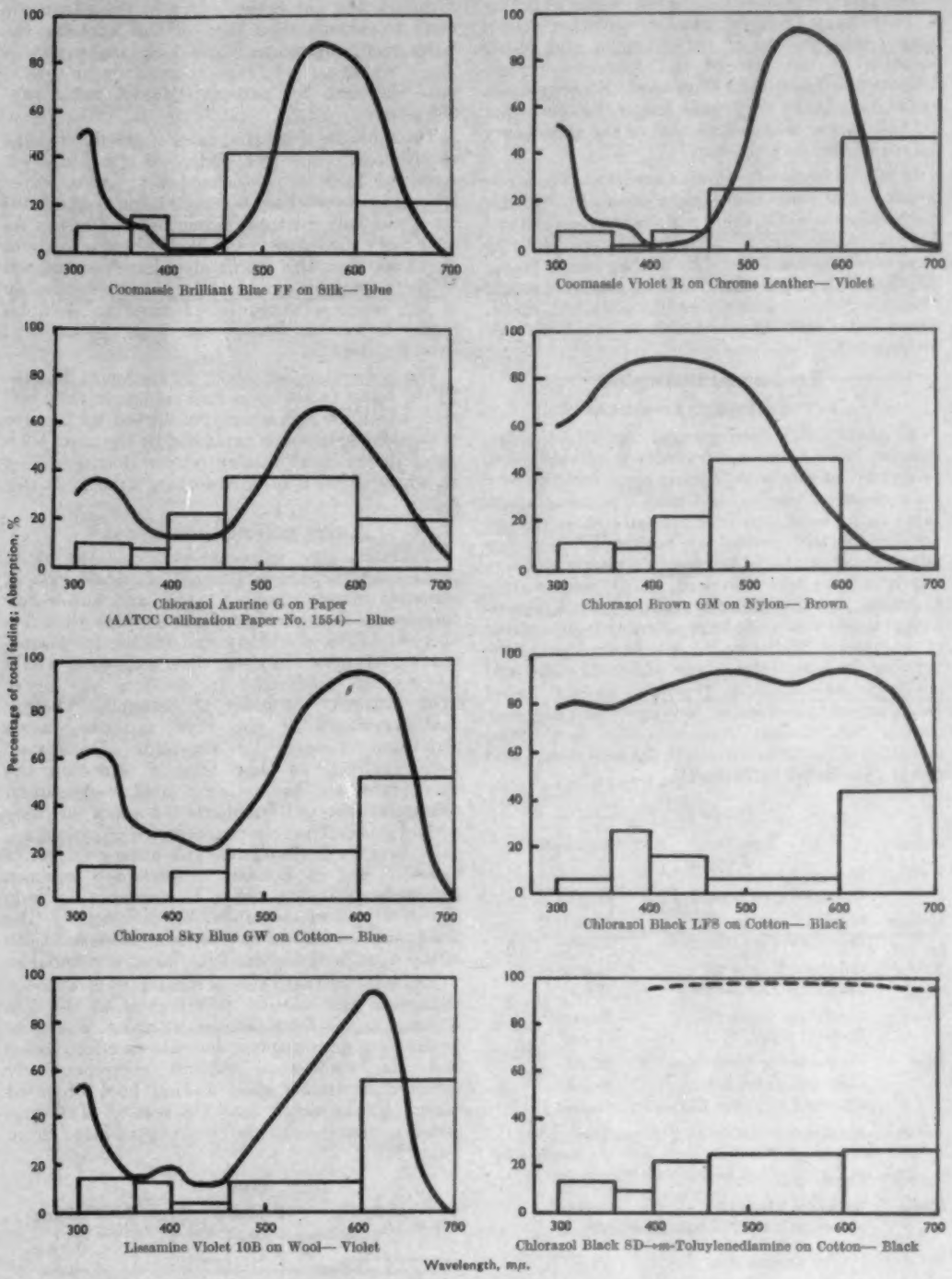
Percentage of total fading; Absorption, %
Coomassie Brilliant Blue FF on Silk— Blue
Coomassie Violet R on Chrome Leather— Violet
Chlorazol Azurine G on Paper
(AATCC Calibration Paper No. 1554)— Blue
Chlorazol Brown GM on Nylon— Brown
Chlorazol Sky Blue GW on Cotton— Blue
Chlorazol Black LFS on Cotton— Black
Lissamine Violet 10B on Wool— Violet
Chlorazol Black SD—m-Toluylenediamine on Cotton— Black
Wavelength, mμ.
Absorption curve of dye solution
Absorption curve of dyed pattern
Proportion of total fading of dyed pattern due to given spectral region

FIG. 3— Diagrammatic Representation of the Contribution to Fading of Five Spectral Regions of Daylight together with the Absorption Spectrum of the Dye (Fugitive Dyes)

This method of determining the degree of fading is only very rough; greater accuracy could have been achieved if the Kubelka and Munk equation or the best of the American colour-difference formulae had been used. Such methods would have taken very much longer, however, and did not appear essential in view of the elementary nature of the investigation.

In the interests of complete accuracy it must be pointed out that the figures obtained by this method for each of the four regions between 295 and 600 mμ. apply only when the pattern is being irradiated simultaneously by all the longer wavelengths. It was not felt, however, that this would in any way invalidate the results obtained, and it would have been impracticable to eliminate this discrepancy.

Results and Discussion

RANDOM FUGITIVE SAMPLES

As about 3500 homogeneous organic colouring matters have been commercially available, comprising about nine main dyeing classes and twenty main chemical classes, and each dye being applicable to at least two or three substrates (to say nothing of the anomalous behaviour of a few mixtures or of the many possible aftertreatments which affect light fastness), it is immediately apparent that a systematic representative investigation would require an impracticably large number of samples. Therefore a selection was made, varying the hue, dyeing type, chemical class, and substrate as widely as possible; for the initial examination, patterns of low light fastness were chosen for obvious reasons. The first series consisted of patterns dyed with the acid, basic, and direct dyes listed in Table IV.

Table IV

Colour	Dye	Substrate
Red	Benzopurpurine 4B	Cotton
	Chlorazol Fast Scarlet G	Linen
Orange	Chlorazol Orange PO	Linen
	Coomassie Fast Orange G	Paper
Yellow	Chlorazol Yellow 2G	Cotton
	Lissamine Flavine FF	Wool
Green	Disulphine Green B	Paper
	Brilliant Green Y	Cotton
Blue	Coomassie Brilliant Blue FF	Silk
	Chlorazol Azurine G	Paper
	Chlorazol Sky Blue GW	Cotton
Violet	Lissamine Violet 10B	Wool
	Coomassie Violet R	Chrome leather
Brown	Chlorazol Brown GM	Nylon
Black	Chlorazol Black LF	Cotton
	Chlorazol Black SD diazotised and developed with *m*-toluylenediamine	Cotton

The fading diagrams are given in Fig. 2 and 3, which include also the absorption curves of the dyes dissolved in water, except for Chlorazol Black SD diazotised and developed on the fibre, in which case the absorption curve for the pattern is given. Although the absorption curve of the pattern is more important than that of the solution, the latter was preferred as it could be measured on a Carey recording spectrophotometer down to 300 mμ., the limit for patterns (Hardy) being only 380 mμ.

These results show that each of the five regions contributes to the total fading if it is absorbed. Even red light is photochemically active, wavelengths greater than 600 mμ. being responsible for about half the total fading in some cases. As none of the dyes examined absorbed above about 660 mμ., this fading must have been caused by quite a narrow band and the quantum efficiency of the reaction would be expected to be quite high— at least by the low standards applicable to dyed textiles.

The anomalous behaviour of Lissamine Flavine FF in being faded by radiations longer than 460 mμ., which are not strongly absorbed by the dye in aqueous solution, is explained by the absorption curve of the dyed pattern (shown dotted in Fig. 2), which shows it to be absorbing strongly in this region.

LIGHT FASTNESS STANDARDS

Although the patterns examined in these preliminary exposures covered a wide range of chemical classes, dyeing types, and substrates, they were all of low light fastness— less than 3— and it would obviously be unwise to assume that conclusions based on these exposures would be equally applicable to patterns of higher light fastness. In order to determine whether the magnitude of the light fastness was a significant factor, the standards themselves[3] were exposed, as they form a series on the same substrate having very similar absorption characteristics, and Standards 1–6 are of the same dyeing class. The results are shown diagrammatically in Fig. 4, Standards 1–3 being exposed in Cheshire, 4–7 in Bombay. Standard 8 was not included, as it has never been known to fade sufficiently for accurate measurement. The absorption curves in Fig. 4 are of aqueous solutions of the dyes used for the light fastness standards.

Fig. 4 shows that there is a marked, progressive change in the relative effectiveness of the five regions: as the light fastness increases from 1 to 7, the long-wave regions decrease in effectiveness and the short-wave regions correspondingly increase, a similar effect having been observed earlier by Luszczak and Zukriegel[4]. The progressive change can be best appreciated from Table V.

Table V

Standard No.	Proportion (%) of Total Fading caused by Radiation—	
	> 600 mμ.	< 460 mμ.
1	36	24
2	26	50
3	19	73
4	4	91
5	5	71
6	0	95
7	0	100

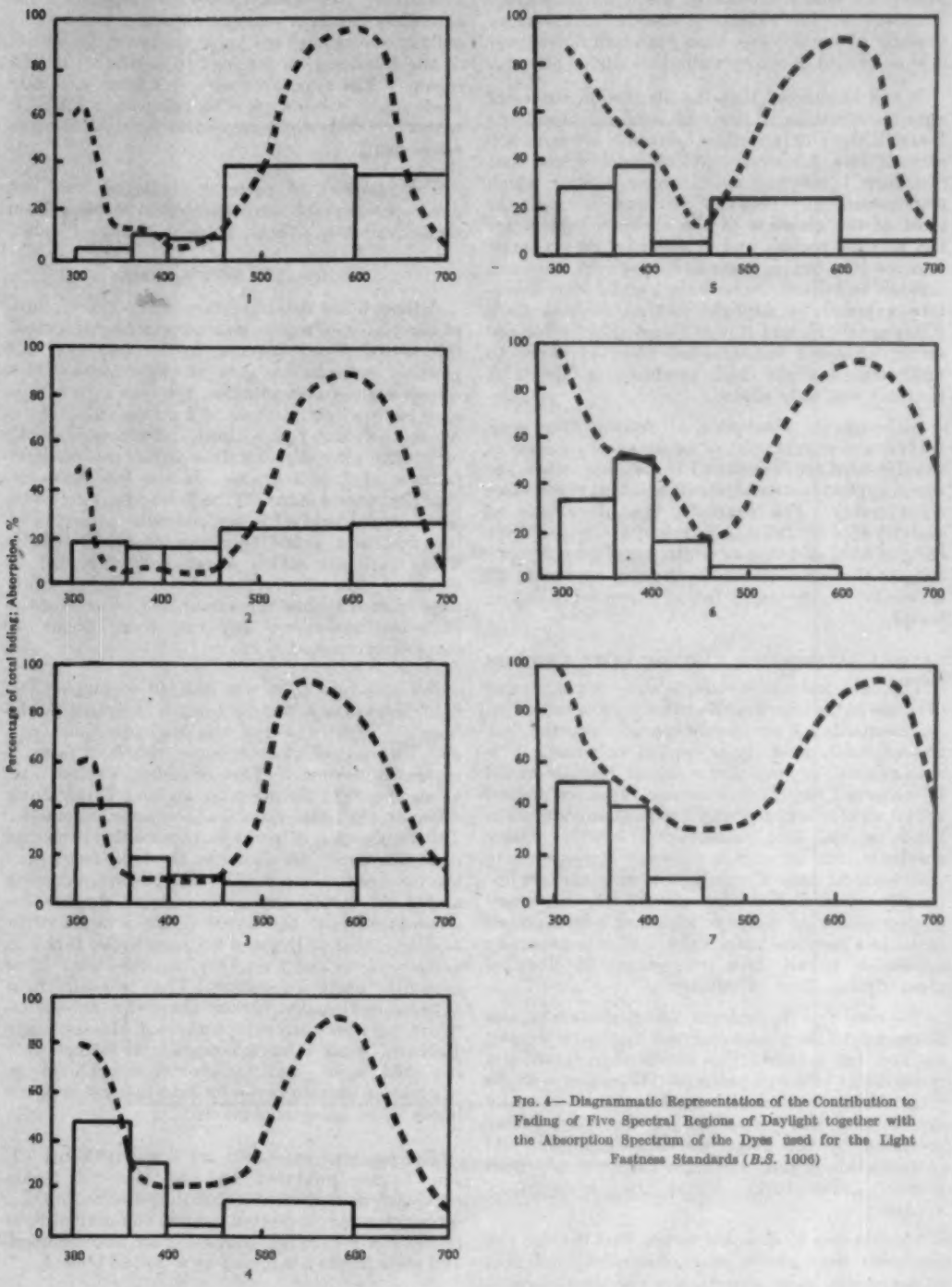

FIG. 4— Diagrammatic Representation of the Contribution to Fading of Five Spectral Regions of Daylight together with the Absorption Spectrum of the Dyes used for the Light Fastness Standards (*B.S.* 1006)

This effect is obviously independent of the absorption characteristics of the dyed standards. Standard 6, for example, absorbs much more strongly above 600 mμ. than Standard 3 does, yet it is not faded at all by radiations above 600 mμ.

It will be noticed that the absorption curves of aqueous solutions of the dyes used for Standards 1 and 2 show only slight absorption between 350 and 450 mμ. Luszczak and Zukriegel [4] found that Standard 1 was not faded under a filter which transmitted only between 340 and 390 mμ. As most of the emission of the enclosed carbon arc lies in that region, and Standard 1 is the most fugitive in a fading lamp as in daylight, this was difficult to believe. Standards 1 and 2 were therefore exposed to daylight behind such a filter (Chance OV 1), and it was found that fading did occur, although the amount when exposure to unfiltered daylight had produced a Grade 1 contrast was only slight.

Although the absorption at 385 mμ. (the peak carbon arc wavelength) of an aqueous solution of the dye used for Standard 1 is very low, when the dye is applied to a substrate its absorption increases considerably. For example, the absorption of undyed wool at 385 mμ. is raised by dyeing from 65% to 85% and that of white paper from 20% to 48% at the same visual depth; this undoubtably accounts for the rapid fading observed in fading lamps.

RANDOM PATTERNS OF VARYING LIGHT FASTNESS

The light fastness standards show a progressive decrease in the effectiveness of long-wave radiation. As Standards 1–6 are merely specially selected, but conventional, acid dyes applied to wool, it is reasonable to suppose that a similar decrease would be observed for other blue acid dyes, or indeed acid dyes of other hues which possess an absorption band in the long-wave region of the visible spectrum, viz. all except yellows. Moreover, any one chemical class of organic colouring matters has usually yielded dyes for several of the many dyeing classes, so therefore what has been found to occur in a restricted range might also be generally applicable to all dyes irrespective of chemical class, dyeing class, or substrate.

To test this hypothesis using the composite filters would have taken far too long, so a simpler method was devised. This consisted in exposing a much wider range of patterns (117) under a single filter which divided the spectrum into two: by determining the percentage of the total fading produced by the longer-wavelength portion and plotting this against the light fastness, any such general relationship would be immediately apparent.

The obvious filter would be one that divided the spectrum into visible and ultraviolet, but this choice would have been illogical, as the difference between the two arises solely from a characteristic of the adult human eye. Instead, therefore, yellow Perspex (ii, Table I) was chosen, as this divided the spectrum at the point (460 mμ.) where the radiation of the enclosed carbon arc passes from the excessively rich ultraviolet–violet region to the extremely deficient region comprising blue, green, yellow, orange, and red light; the latter region will in the following be referred to as the "blue–red region". The exposures with this filter were also made under window glass to bring them into line with normal daylight exposures for light fastness assessment.

The majority of patterns employed were not dyed specially but were selected at random from those available, yellows, however, being excluded.

"NORMAL" LIGHT FASTNESS

Although the determination of the effectiveness of the blue–red region was quite straightforward, the actual light fastness to be employed for plotting purposes was not: the light fastness of a dye as such is a meaningless term, as a given dye may have a light fastness of 2 on one substrate, 5 on another, and 7 on a third; differences in depth of dyeing may also produce differences in light fastness of 2 or 3 grades. It was felt, however, that the general nature of the fading diagram would be governed solely by the molecular structure of the dye, and would therefore be unaffected by those variables which affect light fastness; if, therefore, the effectiveness of the blue–red region were plotted against the actual light fastness of the patterns themselves, any real trend might be completely masked.

For this reason, it was decided to employ the light fastness of a standard depth of dyeing on the fibre for which the dye was originally marketed, and, for want of a better name, this light fastness is termed "normal". This, of course, implies that, when the light fastness on another substrate is different, the latter value is abnormal or anomalous. This implication is, perhaps, reasonable in the case of textile dyes: for example, the light fastness of the vat dye Caledon Red Violet 2RN is 8 on cotton and 2 on nylon, and no-one would dispute the assumption that the nylon figure is anomalous; similarly, that of Duranol Brilliant Violet B is 4 on acetate rayon and 1 on Terylene, the latter figure evidently being anomalous. The "normal" light fastness is usually higher than the anomalous figure, but there are exceptions: e.g. the basic dye Brilliant Green Y has a normal light fastness of 1 (on silk, wool, and tannin-mordanted cotton), whereas on acetate rayon its light fastness is 5, the latter figure being anomalous.

The patterns examined are listed in Table VI. The fading produced by the blue–red region expressed as a percentage of the total fading, i.e. ultraviolet–red, is plotted against the normal light fastness in Fig. 5; Standards 1–7 are also included, and their points are joined by a dotted line.

Fig. 6 and 7 show the actual filter assembly used together with a representative selection of patterns (Fig. 6) and Standards 1–7 (Fig. 7) after exposure behind it.

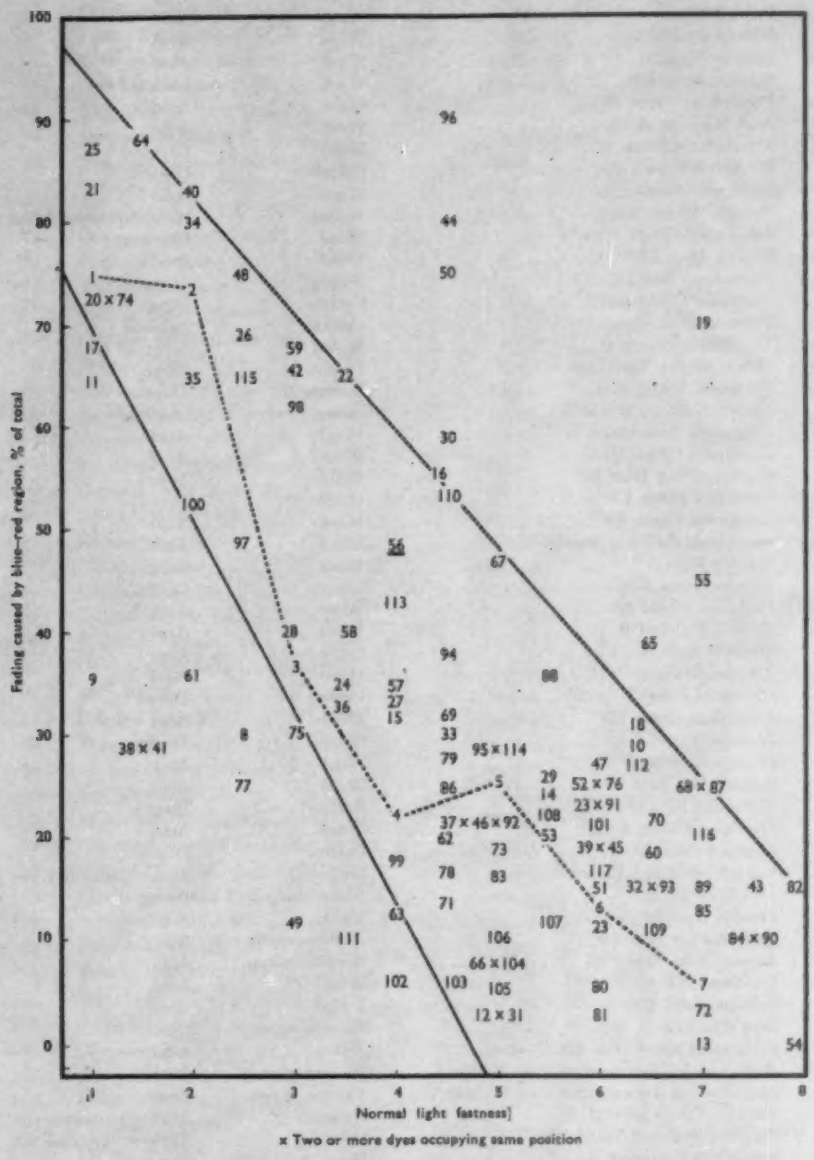

Fig. 5—Effectiveness of the Blue–Red Region as a Function of the Normal Light Fastness

Table VI

Pattern No.	Dye (All ICI unless otherwise stated)	Substrate	Dyeing Class
1	Acilan Brilliant Blue FFR (FBy)	Wool	Acid
2	Acilan Brilliant Blue FFB (FBy)	Wool	Acid
3	Coomassie Brilliant Blue R	Wool	Acid
4	Polar Blue G	Wool	Acid
5	Solway Blue RN	Wool	Acid
6	Solway Blue 2G	Wool	Acid
7	Soledon Blue 4BC	Wool	Indigoid vat
8	Disulphine Green B	Wool	Acid
9	Acid Magenta A	Wool	Acid
10	Solochrome Black WDFA	Wool	Chrome
11	Bismarck Brown GN	Cotton	Basic
12	Lissamine Violet 2R	Wool	Acid
13	Caledon Violet XBN	Nylon	Anthraquinonoid vat
14	Solochrome Dark Blue B	Wool	Chrome
15	Solway Blue PFN	Wool	Acid
16	Durindone Red 3B	Nylon	Indigoid vat
17	Chlorazol Green BN	Cotton	Direct
18	Thionol Ultra Green B	Cotton	Sulphur
19	Durindone Brown G	Nylon	Indigoid vat
20	Chlorazol Sky Blue GW	Cotton	Direct
21	Chlorazol Violet WB	Cotton	Direct
22	Dispersol Diazo Black B	Acetate rayon	Disperse
23	Coomassie Fast Black B	Wool	Acid
24	Lissamine Green V	Wool	Acid
25	Chlorazol Sky Blue FF	Cotton	Direct
26	Chlorazol Black LF	Cotton	Direct
27	Lissamine Green SF	Wool	Acid
28	Coomassie Brilliant Blue R	Wool	Acid
29	Solway Blue G	Wool	Acid
30	Durazol Blue 2GN	Cotton	Direct
31	Carbolan Violet 2R	Wool	Acid
32	Solway Rubinol R	Wool	Acid
33	Durazol Blue G	Cotton	Direct
34	Lissamine Violet 10B	Wool	Acid
35	Chlorazol Diazo Blue 3G	Cotton	Direct
36	Lissamine Green BN	Wool	Acid
37	Durazol Violet 2B	Cotton	Direct
38	Disulphine Blue VN	Wool	Acid
39	Solway Sky Blue B	Wool	Acid
40	Chlorazol Blue G	Cotton	Direct
41	Disulphine Blue AN	Wool	Acid
42	Durazol Grey RG	Cotton	Direct
43	Caledon Brilliant Violet R	Cotton	Anthraquinonoid vat
44	Icyl Blue 2R	Viscose rayon	Direct
45	Solacet Fast Green 2G	Nylon	Acid
46	Solacet Fast Blue 2B	Acetate rayon	Acid
47	Solacet Fast Green 2G	Acetate rayon	Acid
48	Coomassie Green T	Wool	Acid
49	Solacet Navy Blue G	Nylon	Acid
50	Icyl Blue G	Viscose rayon	Direct
51	Coomassie Navy Blue 2RN	Nylon	Acid
52	Brenthol AS–Brentamine Fast Black K	Viscose rayon	Azoic
53	Brenthol AS–Brentamine Fast Corinth V	Viscose rayon	Azoic
54	Caledon Red Violet 2RN	Nylon	Anthraquinonoid vat
55	Caledon Brilliant Violet 3B	Cotton	Anthraquinonoid vat
56	Solochrome Azurine B	Wool	Chrome
57	Rufasol Black B	Nylon	Acid
58	Naphthalene Green G	Wool	Acid
59	Chlorazol Black E	Cotton	Direct
60	Alcian Blue 8G	Viscose rayon	Alcian
61	Disulphine Blue FFN	Wool	Acid
62	Dispersol Diazo Black 2B	Nylon	Disperse
63	Dispersol Diazo Black A	Nylon	Disperse
64	Coomassie Brilliant Blue FF	Paper	Acid

TABLE VI (*contd.*)

Pattern No.	Dye (All ICI unless otherwise stated)	Substrate	Dyeing Class
65	Caledon Green BN	Cotton	Anthraquinonoid vat
66	Duranol Blue Green B	Nylon	Disperse
67	Coomassie Fast Grey 3G	Nylon	Acid
68	Caledon Brilliant Violet 3R...	Viscose rayon	Anthraquinonoid vat
69	Naphthalene Dark Green A	Wool	Acid
70	Durazol Rubine B	Viscose rayon	Direct
71	Dispersol Diazo Black B	Nylon	Disperse
72	Caledon Violet XBN	Nylon	Anthraquinonoid vat
73	Carbolan Violet 2R	Nylon	Acid
74	Chlorazol Sky Blue GW	Cotton	Direct
75	Disulphine Green B	Wool	Acid
76	Duranol Brilliant Violet BR	Nylon	Disperse
77	Soluble Paper Blue 3M	Paper	Acid
78	Duranol Brilliant Blue BN	Terylene	Disperse
79	Durazol Blue 2G	Paper	Direct
80	Lissamine Fast Violet 2B	Paper	Acid
81	Solway Blue 2G	Paper	Acid
82	Caledon Blue GCP	Cotton	Anthraquinonoid vat
83	Naphthalene Fast Orange 2G	Wool	Acid
84	Caledon Dark Brown 6R	Cotton	Anthraquinonoid vat
85	Caledon Brown 3G	Cotton	Anthraquinonoid vat
86	Solway Blue G	Paper	Acid
87	Caledon Khaki R	Cotton	Anthraquinonoid vat
88	Caledon Grey 3B	Cotton	Anthraquinonoid vat
89	Caledon Brown G	Cotton	Anthraquinonoid vat
90	Caledon Brown R	Cotton	Anthraquinonoid vat
91	Carbolan Brilliant Green 5G	Nylon	Acid
92	Duranol Blue 2G	Nylon	Disperse
93	Caledon Grey M	Cotton	Anthraquinonoid vat
94	Thionol Indigo Blue 4R	Cotton	Sulphur
95	Coomassie Fast Orange G	Paper	Acid
96	Icyl Violet BN	Viscose rayon	Direct
97	Disulphine Blue VN	Wool	Acid
98	Chlorazol Diazo Bordeaux 2B	Cotton	Direct
99	Duranol Brilliant Violet B	Acetate rayon	Disperse
100	Lissamine Green SF	Wool	Acid
101	Solacet Fast Blue 2B	Nylon	Acid
102	Solacet Violet R	Nylon	Acid
103	Solacet Fast Red 5BG	Nylon	Acid
104	Coomassie Ultra Sky SE	Nylon	Acid
105	Carbolan Blue B	Nylon	Acid
106	Carbolan Blue R	Nylon	Acid
107	Carbolan Brilliant Blue 2R	Nylon	Acid
108	Duranol Blue 2G	Nylon	Disperse
109	Caledon Green 7G	Cotton	Anthraquinonoid vat
110	Durindone Pink FF	Nylon	Indigoid vat
111	Solacet Fast Violet 4R	Nylon	Acid
112	Caledon Brilliant Red 3B	Nylon	Anthraquinonoid vat
113	Durazol Blue 4G	Cotton	Direct
114	Solway Celestol B	Silk	Acid
115	Chlorazol Orange PO	Linen	Direct
116	Caledon Jade Green XBN	Cotton	Anthraquinonoid vat
117	Carbolan Green G	Nylon	Acid

Although the degree of scatter in Fig. 5 is considerable (as was expected), the majority of points fell into a well defined main sequence which slopes steeply, indicating that the contribution made by the blue–red region does generally decrease as the normal light fastness increases.

It was not intended to investigate the dyes which lie outside the main sequence, but it was noticed that those grouped together showed certain similarities: for example, No. 8, 9, 38, 41, 61, 77 are triphenylmethane derivatives, and No. 44, 50, 96 are direct azo dyes specially synthesised for application to viscose rayon.

THEORETICAL CONSIDERATIONS

All the dyes indicated in Fig. 5 possess an absorption band in the blue–red region, yellows being deliberately excluded; the efficiency of such radiation, however, clearly varies considerably, being very high for fugitive dyes and zero for a number of dyes of high normal light fastness. Undoubtedly, the intensity and the width of the absorption band in the blue–red region will affect the efficiency as determined by these simple experiments, but when a series of dyes of similar absorption spectra are examined, as in Fig. 4, it is apparent that another factor is involved, as the efficiency in the case of Standard 6 is very much lower than in the case of Standard 1, yet the band of the former is somewhat wider than that of the latter.

There is one related observation recorded in the literature: Vraneck [5] found that cobaltioxalates in solution possess two absorption bands— one in the red and one in the blue— but photochemical degradation occurs only with blue light, not with red. Similar complete inactivity of the absorption band in the blue–red region would therefore be the simplest explanation of the results given in Fig. 5, intermediate efficiencies also occurring if the short-wave absorption band extended beyond 460 mμ. Although this could be a contributory factor in many cases, it cannot account for the results given in Fig. 4, as overlapping does not occur and yet intermediate efficiencies are observed in the case of Standards 3, 4, and 5 (Table V).

This variation in efficiency could be explained if only part of the absorbed radiation in the blue–red region were active; that is to say if, within a single absorption band, only certain wavelengths were capable of causing fading. If this were true, then it would be unlikely that wavelengths would be active or inactive at random; it would be more likely, however, that, within one absorption band, the longest radiation is inactive and the shortest radiation active, as the latter results in the absorption of more energy than the former. This would imply, therefore, that for such dyes there exists a critical wavelength such that radiation of shorter wavelength than this critical value is photochemically active if absorbed, whereas radiation of longer wavelength is inactive even if strongly absorbed.

Table VII

Filter	Region transmitted (mμ.)
(vi) Chance OX 5	>800
(vii) Chance OV 1 glass + red Perspex ...	>690
(viii) Chance OX 7 glass + red Perspex ...	>650
(i) Red Perspex	>600
(ix) Orange Perspex	>530
(x) Chance OY 4 glass	>475
(ii) Yellow Perspex + ultraviolet absorber	>460
(iii) Cellulose acetate + ultraviolet absorber	>400
(iv) Perspex + ultraviolet absorber ...	>360
(v) Perspex clear	>295

In order to test this hypothesis, some exposures were made behind a second composite filter, which divided the visible spectrum into eight regions, as shown in Table VII. A number of patterns were exposed under these filters until there was complete destruction of the dye under the clear Perspex. The critical wavelength was then assumed to lie between the region of longest wavelength where fading, however slight, occurred and the one beyond, where fading was prevented.

Table VIII

Dye	Faded by— Radiation longer than (mμ.)	Not faded by— longer (mμ.)	Absorption ceases at (mμ.)
Victoria Pure Blue BON	690	800	745
Acilan Brilliant Blue FFR (FBy)	650	690	685
Chlorazol Brown GM ...	600	650	625
Magenta PN	600	650	620
Auramine O	475	530	485
Acronol Yellow TC ...	460	475	465

With fugitive dyes it was found that fading ceased when absorption ceased, as shown in Table VIII. In such cases, the "critical" wavelength has no special meaning, as the wavelength at which fading ceases is undoubtedly that at which absorption falls to zero. For many dyes of higher normal light fastness, however, there is a critical wavelength above which absorbed radiation is photochemically inactive; some examples of this are given in Table IX.

Table IX

Dye	Critical Wavelength between (mμ.)	Colour of Inactive Absorbed Radiation
Durindone Blue 4BC	475 and 530	Green, orange, red
Durazol Red 6B ...	475 and 530	Green and orange
Solacet Violet B ...	475 and 530	Green and orange
Durazol Green 5G ...	460 and 475	Orange and red
Caledon Violet XBN	400 and 460	Blue, green, orange, red

It is possible, therefore, that the position of the critical wavelength is the chief factor governing the effectiveness of the absorption band in the blue–red region. Since this decreases as the normal light fastness increases (Fig. 5), the obvious inference is that the critical wavelength moves towards the ultraviolet as the normal light fastness increases,

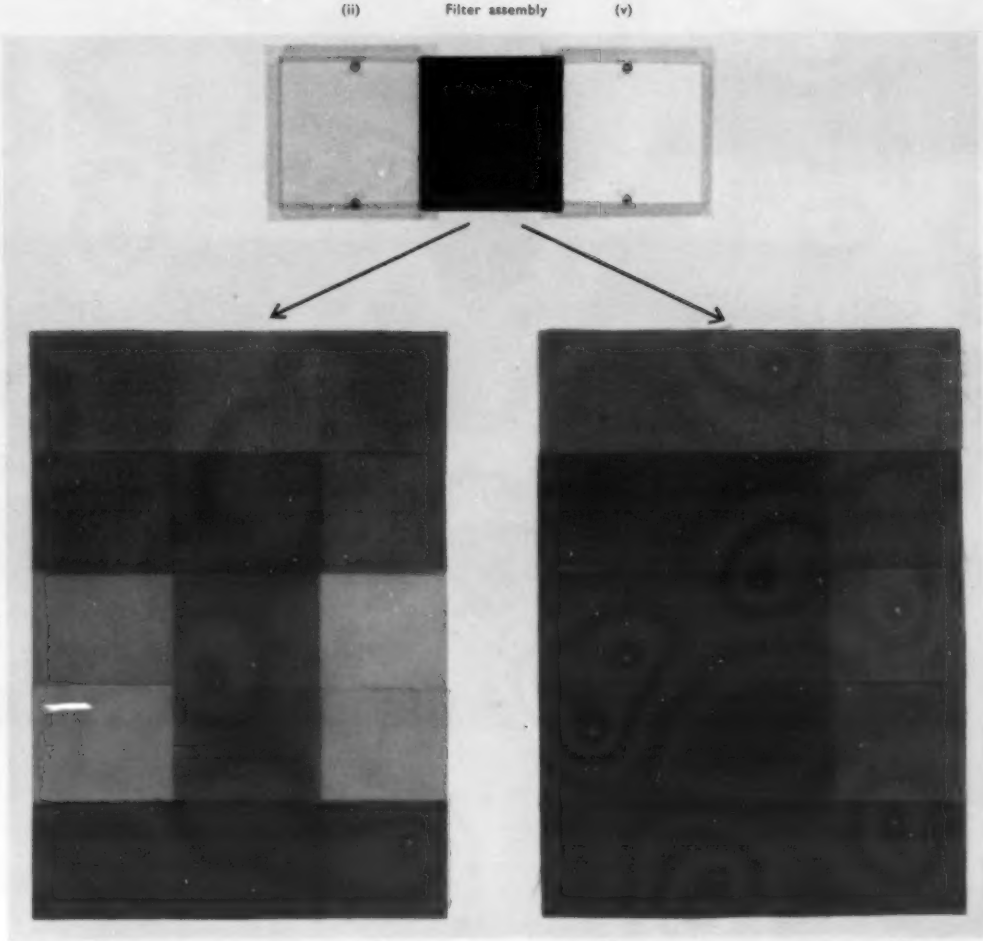
(ii) Filter assembly (v)
115—Chlorazol Orange PO on linen
27—Lissamine Green SF on wool
64—Coomassie Brilliant Blue FF on paper
21—Chlorazol Violet WB on cotton
59—Chlorazol Black E on cotton
83—Naphthalene Fast Orange 2G on wool
117—Carbolan Green G on nylon
104—Coomassie Ultra Sky SE on nylon
111—Solacet Fast Violet 4R on nylon
71—Dispersol Diazo Black B on nylon
(ii) (v)
Patterns of Low Normal Light Fastness
(ii) (v)
Patterns of High Normal Light Fastness

FIG. 6— Typical Patterns of Low and High Normal Light Fastness exposed behind Yellow (ii) and Clear (v) Perspex

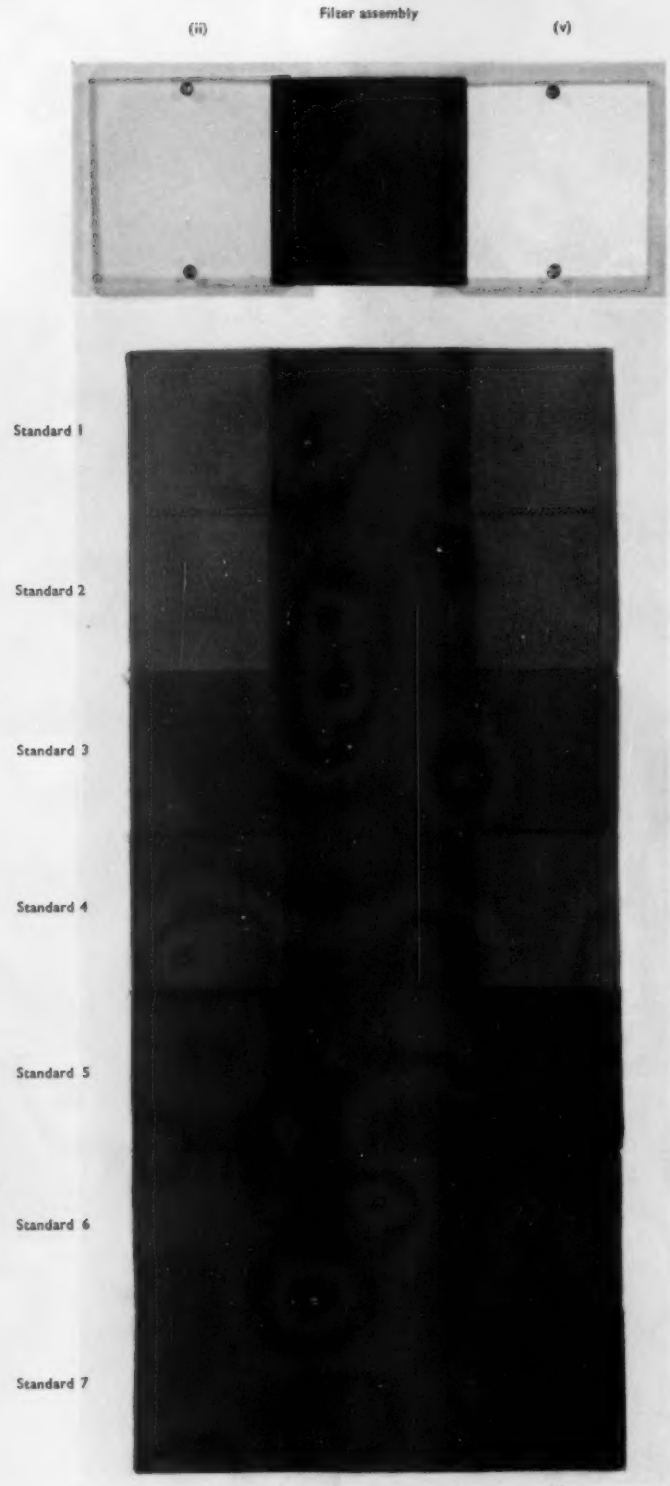

FIG. 7— Light Fastness Standards (*B.S.* 1006) exposed behind Yellow (ii) and Clear (v) Perspex

and that if the critical wavelength had been determined and plotted against the normal light fastness a result similar to that shown in Fig. 5 would have been obtained.

This hypothesis, of course, implies that dyes of high light fastness are sensitive to a smaller fraction of solar radiation than are fugitive dyes of the same hue. This decrease in sensitivity alone, however, cannot account for the range of light fastness values encountered; for, if the critical wavelength lies in the green, only about half the radiation is inactive, and this can account only for a doubling of the resistance to fading. Standard 6 is, however, thirty-two times as resistant as Standard 1.

As there is a definite amount of energy associated with each wavelength*, the critical wavelength would appear to be a measure of the energy level of the lowest excited state which is photochemically active; this is generally believed to be the relatively long-lived triplet state.

It is not suggested, however, that all radiations shorter than the critical wavelength are equally effective if equally absorbed, or in other words, that the quantum efficiency is independent of wavelength throughout the region below the critical wavelength. It is more reasonable to assume that the quantum efficiency decreases with increasing wavelength, and that the critical wavelength is merely the point at which it falls to zero. The appearance of the patterns exposed under the second composite filter tended to confirm this, as in no case was there marked fading under one filter and no fading under the one above; instead, the amount of fading decreased steadily, making determination of the point at which fading ceased somewhat uncertain, as one felt that more prolonged exposure might have resulted in slight fading. This appearance would, of course, occur if the quantum efficiency approached zero asymptotically.

PRACTICAL CONSIDERATIONS

This critical wavelength theory enables one to predict the regions causing fading of a dyeing from a knowledge of—

(*a*) The absorption characteristics of the dye.

(*b*) Its normal light fastness.

From this knowledge it is possible to make interesting and important deductions concerning—

(1) The spacing of the light fastness standards when exposed to different light sources

(2) Those patterns whose "lamp" fastness ratings will be significantly different from their daylight ratings solely because of the differences in the composition of the photochemically active radiation, viz. 295–800 mμ.

(3) The dangers inherent in the Standard Fading Hour concept.

Each of these will be discussed in detail.

* e.g. 38 kcal. for radiation of 750 mμ. and 90 kcal. for radiation of 300 mμ.

(*1*) *Spacing of the Standards*

Fig. 5 shows that Standards 1 and 2 are much more sensitive to radiation longer than 460 mμ. than are the remaining standards, and that Standard 4 is faded mainly by radiation shorter than 460 mμ., viz. violet and ultraviolet. If Standards 1–4 are exposed to different light sources so as to produce the same small fade on Standard 4, it can be assumed that the same amount of radiation shorter than 460 mμ. will be needed irrespective of the light source. In daylight this quantity of ultraviolet radiation and violet light will be accompanied by a much greater amount of blue–red light, which will produce considerable fading on Standards 1 and 2; in a lamp, however, this same amount of ultraviolet and violet will be accompanied by a much smaller amount of blue–red light, and therefore less fading of Standards 1 and 2 will occur. Standards 1–4 will therefore be more closely spaced in a fading lamp than they are in daylight; Fig. 8 shows that this is so, the standards being closest together when the blue–red region is completely excluded with a Chance OV 1 filter.

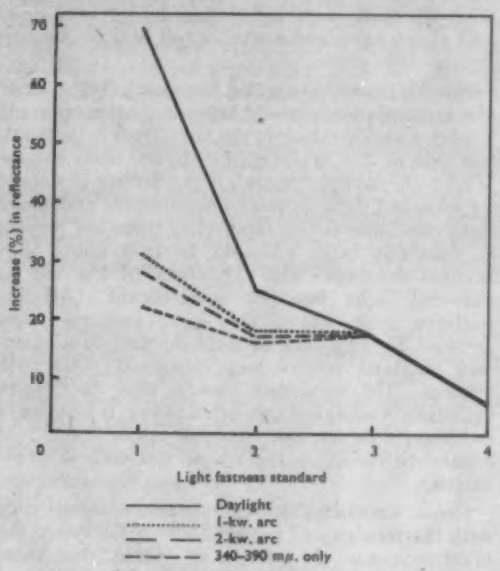

Fig. 8—Spacing of Standards 1–4 when exposed to Different Light Sources

American Standards—It has been claimed[6] that the spacing of the American standards in the lamp is closer to the spacing in daylight than is the case with the British standards. This would be expected, as the most fugitive American standard, L 2, is equal in light fastness to British standard 4, and consequently the effectiveness of the blue–red region in causing fading is much less; it is No. 56 (underlined in Fig. 5), and the contribution of the blue–red region in daylight is 49%, compared with 76% for the most fugitive British standard. If the American standards covered as wide a range of light fastness as the British, they would in all probability show similar discrepancies in spacing.

(2) *Fading Lamp Anomalies*

Fig. 5 shows that the seven light fastness standards fall inside the main sequence, and it would appear that anomalies in assessment due to differences in emission characteristics will be least with patterns which also fall within the main sequence and greatest with patterns which fall well outside.

Considering first patterns which lie below the main sequence, these are of light fastness below 3 and are faded mainly by ultraviolet and violet light, whereas the relevant standards are faded mainly by blue–red light. As the lamp is very rich in ultraviolet and violet light, such patterns will be rated lower in the lamp than in daylight. This effect will be most prominent with dyes of normally high light fastness, applied to substrates which considerably reduce the light fastness; thus Caledon Violet XBN (No. 72 in Fig. 5) has a normal light fastness of 7 on cotton, but on nylon its fastness in daylight is 3 and its fastness in a lamp only 1.

Similar behaviour is shown by Durazol Red 2B aftertreated with a cationic dye-fixing agent: this combination is faded mainly by ultraviolet and violet light, and a similar fall in light fastness occurs.

Similar reasoning applied to patterns lying above the main sequence would suggest that they would receive a higher rating in the lamp than in daylight, but this, in fact, is not so, for the following reason. Whilst in daylight most of the fading is caused by blue–red light, ultraviolet radiation and violet light can cause fading, and when these are present in relatively large amounts, as in a lamp, they become dominant and the effect of the loss of blue–red light becomes insignificant. All the patterns lying above the main sequence were assessed by exposure to daylight and in a lamp, and identical results were obtained with both sources. The important fact is that short-wave radiation if absorbed can cause fading if long-wave radiation does so; the converse is not true if the long-wave radiation lies above the critical wavelength.

Gross anomalies should therefore occur only with the minority of patterns which fall below the main sequence. It follows, of course, that these could be eliminated if Standards 1–3 also were faded mainly by violet and ultraviolet radiation. However, there are unlikely to be any blue dyes with affinity for wool which would be of this type and of the required light fastness.

(3) *The Standard Fading Hour Concept*

The method of assessing light fastness most widely used in the U.S.A. is to determine the number of Fade-Ometer hours necessary to produce an easily perceptible fade on the pattern under test. This method has been refined with considerable care by American workers, who closely specify the operating conditions of the lamp and who provide a calibration paper by which the performance of a lamp can be compared with the performance of a master lamp in the National Bureau of Standards in Washington. Unfortunately, however, now that the importance of the spectral composition of the light source is known, it would appear that this system contains two inherent defects.

The first of these arises from the fact that the light-sensitive calibration paper, which is dyed with a direct dye having a light fastness of 1, is faded mainly by the green–red region in daylight, the complete fading diagram being given in Fig. 3. Because the dye does not absorb strongly in the region where the emission of the enclosed carbon arc is greatest, it cannot be particularly sensitive to changes in arc output in this region, which are so important because they cause most of the fading. Moreover, because the paper is much more sensitive to the green–red region it is possible that a severe drop in emission of the violet and ultraviolet peaks could be balanced by a simultaneous increase in the emission in the green–red region. It is known that the balance of radiation is very sensitive to air leaks between the globe and its seating, which are very difficult to control with any degree of accuracy.

It must be admitted that this fear may be groundless, as investigators in the U.S.A. have found that, providing the lamps used are running under the specified conditions and that they have been properly calibrated, the variations in determining the number of Standard Fading Hours of a wide variety of patterns in many different lamps are only slight. It should be emphasised that the majority of American lamps are of uniform type, a fact which obviously reduces variations, but that British lamps vary considerably in arc wattage, presence or absence of heat filters, etc.

The second inherent defect is, however, much more important, and arises out of the assumption widely held in the U.S.A. that one standard Fade-Ometer hour can be equated to a definite number of hours of bright sunshine. Not only does the lamp manufacturers' literature encourage this assumption *, but the AATCC Research Subcommittee on Fastness to Light has reported [7] that " . . . . . tests were made to learn how the fading of an average Fade-Ometer hour compares with an average hour of sunlight fading". It can be deduced from Fig. 5, however, that the range of such ratios must be very large, for the following reasons—

Many patterns dyed with dyes of high normal light fastness are faded only by radiations shorter than 460 mμ.; the intensity of this radiation at the pattern position in a fading lamp is much greater than occurs in the brightest sunshine at sea-level (at 385 mμ. it is fourteen times as great [1]). One hour in the lamp will therefore be equivalent to many hours of sunlight exposure. In the case of fugitive blue and violet patterns, however, the efficiency of the predominating radiation in the arc is very low, and therefore one hour in the lamp could conceivably be equivalent to less than one hour's sunlight exposure.

* e.g. "1 hour FDA–R Fade-Ometer equivalent to 1·7 hours summer sunlight in Washington for dyed textiles and 3·3 hours for lithographic and printing inks."

Confirmation of this has been given by Godlove[6], who determined the number of hours of standard sunshine (viz. on clear sunny days between 9 a.m. and 3 p.m. between April 1st and October 1st) needed to produce the same degree of fading as a given number of Standard Fade-Ometer Hours for each of the British light fastness standards. He found that in the case of Standard 1 one hour in the lamp was equivalent to 1·2 hr. of sunshine, whereas with Standard 7 one hour was equivalent to 47·6 hr. of sunshine. He did not publish the ratios for the intermediate standards, but it is highly probable that they would show a steady increase between the two extremes, as it has been established (Fig. 5) that the effectiveness of ultraviolet and violet radiation increases steadily with increasing light fastness. Godlove also determined the sunlight : Fade-Ometer ratio for each of the American standards; these fell between 2·9 and 11·9. The range in this case is smaller partly because the ultraviolet and violet radiation is more effective with L 2 than with British standard 1, but mainly because American standards L 3 to L 8 are each blends of slubbing dyed with the dye used for L 2 with slubbing dyed with the dye used for British standard 8 (Indigosol Blue AGG), which has never been observed to fade, the surface fibres usually breaking away before any fading occurs.

As the ratios of sun hours to Fade-Ometer hours cover such a wide range, it is statistically misleading to determine an average value, for this would apply only to an insignificant minority of patterns.

It must be emphasised that when light fastness determinations are made employing standards—either British or American—these defects disappear and differences in radiation characteristics are minimised, as the standards measure the amount of actinic radiation falling on the pattern under all testing conditions. When standards are used, there is no need for lamp calibration, and lamps employing the light from a 1-kw. arc filtered by two thicknesses of glass and one of water should give the same result as an unfiltered 2-kw. arc (these represent the slowest and the fastest lamps generally used in Great Britain), providing, of course, that pattern temperature and moisture content during exposure are identical.

Conclusions

The actinic radiation emitted by the glass-enclosed carbon arc extends over the same region (330–800 mμ.) as daylight transmitted by window glass. The balance of radiation is quite different, however, the intensity of violet and ultraviolet radiation falling on the pattern during exposure in a lamp being very much greater than that in the brightest daylight at sea-level, whilst the intensity of the blue–green–yellow–orange–red light is very much less. Gross errors in light fastness assessments arising from these differences, however, are confined to a minority of patterns whose daylight fastness is not greater than 4: these will be downgraded to 1 or 2. These differences could, however, result in errors of ½ or 1 grade, which can be extremely important in routine testing, and therefore lamp exposures should never be regarded as a substitute for daylight testing but merely as a means of obtaining a fairly reliable indication of the actual light fastness when time does not allow a daylight exposure to be made.

It would be easy to eliminate these anomalies completely by interposing a pale yellow filter between the arc and the patterns, which would reduce the ultraviolet and violet content to the proportions occurring in daylight. It would be quite impracticable to do so, however, as the lamp would then be as slow as a dull summer's day.

In view of the widespread use of carbon-arc fading lamps, it is indeed fortunate that inevitable gross anomalies in light fastness testing will be confined to a minority of patterns. As these are all downgraded to 1 or 2, it would not take long to make a daylight exposure to prove that the lamp assessment is false whenever a disagreement occurs. Even in the depth of winter, one week's exposure is usually sufficient to produce a marked fade on Standard 1, which should immediately cast doubt on the lamp exposure.

The exoneration of spectral differences naturally lends weight to the opinion expressed earlier[1] that most gross anomalies in testing are caused by differences in surface moisture content during exposure. This aspect is at present being actively investigated by the Light Fastness Subcommittee.

* * *

The author wishes to express his gratitude to those members of the Society's fastness Committees and those members of I.C.I. Dyestuffs Division who read the paper in its original draft and who made so many valuable suggestions concerning its presentation.

Dyehouse Department
Imperial Chemical Industries Ltd.
Dyestuffs Division
Hexagon House
Manchester 9

(*Received 17th October 1955*)

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CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

The Effect of Organic Solvents on the Absorption of Dyes by Wool from Aqueous Solutions at Low Temperatures

A number of experiments have been carried out to determine the effect on the uptake of selected wool dyes by wool yarn, felt, and cloth of adding organic solvents to the aqueous dye solution. Certain solvents have been found to produce a marked increase in the rate of dyeing, so that virtually complete exhaustion of the dye solution can be achieved in a relatively short time and at temperatures considerably lower than those required by conventional methods. The results so far obtained seem to be of sufficient theoretical and practical interest to warrant a much more detailed study of this phenomenon, but it was considered desirable to put on record details of some of the results already obtained.

Of the solvents examined, sparingly soluble amines, alcohols, and phenols are most effective. A fairly detailed examination of the effect of *n*-butanol, for example, showed that a number of typical acid dyes and, particularly, neutral-dyeing 2 : 1 dye : metal complex dyes (Irgalans (Gy)) were much more rapidly absorbed from an aqueous solution saturated with butanol than in the absence of the alcohol. Complete exhaustion was obtained with 4% Irgalan Grey BL (calculated on the weight of wool) after dyeing for only 30 min. at 60°C. No significant difference in washing fastness could be detected, by the S.D.C. Washing Test No. 2 for dyed wool materials, between samples so dyed and samples dyed to approximately the same depth by the normal procedure, i.e. dyeing started at 50°C., the temperature raised to 100°C. during 45 min., and dyeing continued at this temperature for 45 min. From this it was inferred that adequate penetration of the fibre had been achieved even at this low temperature and in so short a time of dyeing.

A further investigation was then carried out in which a large number of solvents were examined. It was found that several were as effective as, and others more effective than, 1-butanol in promoting the absorption of the 2 : 1 metal-complex type of dye at low temperatures. Thus when 2% Irgalan Grey BL was applied to wool yarn (5 g.) from an aqueous solution at 50–55°C. in presence of thymol (1 g.), the rate and the extent of dye absorption were such that after 15 min. at least 75% of the dye had been taken up by the wool. This figure is based on visual observation only, and may be much higher. The isomer of thymol, carvacrol, and other cresols were also very effective, as was also the alcohol, geraniol.

The effect of thymol was then examined in more detail. The solubility of thymol in water at 55°C. is 2 g./litre. Thus, when 1 g. of thymol was added to 450 c.c. of water at this temperature, a large amount remained floating on the surface. If the wool yarn was then immersed and agitated in the solution for 1–2 min. before the dye solution (50 c.c.; 0·2% Irgalan Grey BL) was added, very rapid local uptake of dye occurred owing to prior local absorption of the thymol.

A modified technique was therefore adopted in which an aqueous emulsion of the thymol (1 g.) in water (450 c.c.) was prepared. The dry, unscoured yarn was immersed in the emulsion for 1–2 min. at 55°C., the dye solution (50 c.c.) added, and dyeing continued at 50–55°C. for the requisite time. By using an emulsion of those solvents which are only sparingly soluble in water much more uniform absorption of dye was obtained.

It would appear that the effective solvents are first absorbed at the water–fibre interface, and the dye is then taken up via the solvent layer. This would explain the need for uniform initial absorption of the solvent by the fibre. Further, it was found that wool yarn treated in an aqueous emulsion of thymol, rinsed, and then immersed in the dyebath absorbed dye more rapidly than a sample dyed under identical conditions but without such a pretreatment. Similarly, wool yarn impregnated with thymol by immersion in an alcoholic solution followed by evaporation of the alcohol at room temperature also absorbed dye more rapidly than untreated wool. When a 0·05% aqueous solution of Irgalan Grey BL was shaken with an equal volume of thymol at 60°C., the dye was almost completely transferred from the water to the thymol. The resulting solution of dye in thymol shaken with distilled water at 60°C. showed no transfer of dye from the thymol layer to the water layer. Similar results were obtained with the free acid of Cardinal Red J and 1-butanol at room temperature, the aqueous layer becoming completely colourless.

The effect of adding solvents to the dyebath is much more marked with the neutral-dyeing 2 : 1 metal-complex dyes than with any other type so far examined, although a slight increase in the rate of absorption was observed with Polar Red G (Gy) and Carbolan Crimson B (ICI). With the latter the hue obtained on wool in the presence of thymol at 55°C. was much bluer than that obtained by dyeing from an aqueous solution at the same temperature and was much nearer the colour obtained by conventional dyeing at the boil. The absorbed dye must therefore be molecularly dispersed throughout the fibres and not simply mechanically precipitated upon them.

There are no doubt many other solvents which will accelerate dyeing at low temperatures, and their possible technological value need hardly be emphasised to readers of the *Journal*.

C. B. Stevens
L. Peters

Department of Colour Chemistry and Dyeing
Department of Textile Industries
The University
Leeds 2

25th January 1956

Notes

Meetings of Council and Committees
February

Council— 8th

Finance and General Purposes— 1st

Colour Index Editorial Panel— 1st, 15th, and 28th

Publications— 21st

Textbooks and Monographs Subcommittee— 21st

Diplomas— 2nd and 29th

Fastness Tests Co-ordinating Committee— 28th

Society's Perkin— 3rd

Perkin Executive— 3rd

Perkin Main— 14th

Review of Textile Progress— 2nd

Deaths

We regret to report the loss by death of Mr. F. Alcock, Mr. D. W. Marshall, Mr J. K. Steel, and Mr. E. S. Walker.

A.S.D.C. Examination

It is intended to hold an examination for the Associateship of the Society of Dyers and Colourists on Thursday–Saturday, 14–16th June 1956. Completed application forms (obtainable from the Society at 19 Piccadilly, BRADFORD 1, Yorkshire) must reach the Society not later than 31st March 1956.

British Cotton Industry Research Association

Dr. D. W. Hill has succeeded Dr. F. C. Toy as Director of Research.

Linen Industry Research Association

Mr. D. A. Derrett-Smith, a Fellow of the Society, at present Deputy Director of Research, has been appointed Director of Research in succession to Dr. A. J. Turner, who is retiring.

Der Deutsche Färbertag 1956

The sixtieth anniversary of the founding of the Verein Deutscher Färber will be celebrated at the annual conference, to be held this year on July 12–16th in Augsburg. Full details can be obtained from the Secretary of the V.D.F., Dr. K.-H. Möller-Klepzig, Düsseldorf-Oberkassel, Luegallee 68, Germany.

Testing the Colour of Printing Inks

The Printing, Packaging, and Allied Trades Research Association has developed a method, known as the "weighed-shell" technique, which enables a known weight of ink to be laid over a given area of paper under standard conditions, so that any differences in colour seen between the final prints are caused by differences in the inks. The colour of an ink depends only slightly on the type of machine used. Thus with *B.S.*1480 magenta ink, and the same paper and ink film thickness, prints from a Miehle press were less glossy and less saturated than those from a Vandercook proof press. Printing pressure seems to have no appreciable effect on standard prints apart from mottling of thick ink films at high pressure. Work on this test is being conducted in co-operation with several of the Association's members. C. O. C.

Research on Detergents

The Report for 1954–55 of the National Research Council of Canada states that studies of the adsorption of detergents on textiles and their desorption have led to a better understanding of detergent action and have indicated possibilities for obtaining higher efficiencies in laundering.

Bicentenary of the Publication of Home's *Experiments in Bleaching*

In 1756 there was published in Edinburgh a book entitled *Experiments in Bleaching*, by Francis Home. The advertisement (or preface, as we should now term it) to the book stated—

> At the desire of the Honourable Board of Trustees for the improvement of fisheries and manufactures in North Britain, the following treatise was composed, read in different lectures, and the experiments, so far as it was possible, were performed before the bleachers of this country. It is now published in consequence of a petition presented by the bleachers to the Honourable Board of Trustees.

Home's book had a profound influence not only in Britain but also on the Continent, where French and German editions speedily appeared. Subsequently, the Board of Trustees awarded him a gold medal for his work.

Home was the first man to investigate the bleaching of cotton and linen in a systematic manner, and his work still stands as a model of the way in which experiments should be planned and recorded, and how deductions should be drawn from the results. He has very strong claims to be regarded as the founder of textile chemistry.

His work showed conclusively that the ideas held at that time on the mechanism of bleaching, and particularly the part played by the bran or buttermilk then used, were completely erroneous, and that it was far better to replace these materials by sulphuric acid. His work and that of Berthollet in introducing chlorine into bleaching are the two foundation stones on which the modern cotton-bleaching industry is based. It is a matter of pride to the Society that it possesses a copy of the first

edition of this most important book, a copy which has a unique link with the great man who wrote it, for the flyleaf bears the inscription, presumably in Home's own hand— "To the Honble. Board for the Improvement of the Linnen Manufacture of Ireland. By the Author."

At a time when the centenary of Perkin's discovery of the first dye made from coal-tar is being celebrated, the bicentenary of the work of another great British chemist, work which in its own field was also epochmaking, should not be allowed to go unhonoured. C. O. C.

Dyeing of Felt

The British Hat and Allied Feltmakers' Research Association reports that it is to study the fastness of the various classes of dyes on fur felt, and to attempt to ascertain why the wet fastness on fur felt is generally lower than that on wool. It has been found that a lower temperature of dyeing improves the quality of the felt without reducing the fastness. The effect of sequestering agents such as ammonium thiocyanate on felt degradation has been investigated.

Calico Printers and the English Penal Code

According to Arthur Koestler, the revolt of public opinion against the severe penalties imposed by English courts in the early nineteenth century started with two petitions from the proprietors of bleaching-ground establishments and calico printers of London. In these petitions the bleachers and the printers asked that stealing from their establishments should cease to be a capital offence because juries habitually refused to convict the thieves.

Apparent Nearness of Colours

Work in the psychological laboratory at Johns Hopkins University shows that a bright surface appears to be closer to the observer than a dull one, and highly saturated or deep colours generally seem closer than paler colours of low saturation.

Virus for Grub of Clothes' Moth

The Plant Virus Research Unit of the Agricultural Research Council at Cambridge now has a virus which can be sprayed or dusted on clothing and furnishings. It kills the grub, but not the adult moth. Unfortunately, this treatment is not fast to dry cleaning or washing.

Research on Scientific Communication

Work on the use of electronic computing machines to translate scientific papers from one language into another has been done in the U.S.A., notably at the Massachusetts Institute of Technology. It is now reported that the Nuffield Foundation has offered to support work at Birkbeck College, London, on mechanical translation.

The Foundation has also set aside over £40,000 for the support of journals in fundamental subjects. The grant to scientific periodicals will be made, on a yearly basis, in consultation with the Royal Society. No periodical will be supported unless, in the opinion of the allocating committee, it is doing all within its power to make its economic position stable or else is prepared to accept and act upon the advice offered by a panel of advisers. The Foundation is already undertaking a pilot survey of the publishing and distribution practices of the learned periodicals.

Centenary of Indian Patents System

The Indian Patents system became 100 years old on 28th February 1956, the Indian Act No. VI of 1856, which set up the system, having received the assent of the Governor-General on 28th February 1856.

The Indian Government celebrated this event by holding in Calcutta a ten-day exhibition of patented articles, registered designs, and other products of Indian manufacture. Concurrently with the exhibition, a symposium on the patents system was held in Calcutta. To mark the centenary, a special souvenir containing articles on Indian and foreign patent law and practice has been published. C. O. C.

New Books and Publications

Technical Manual and Year Book of the American Association of Textile Chemists and Colorists
Volume 31 — 1955

Edited by W. H. Cady. Pp. 623. New York: Howes Publishing Co. Inc. Price, $6.50 ($6.00 in U.S.A. and Canada); $5.50 and $5.00 to AATCC members.

This volume presents an up-to-date account of the accomplishments and technical contribution of the Association. The reports of the various Research Committees and the details of the AATCC test methods are always a source of interest to British readers and a valuable means of indicating trends of technical development in the U.S.A. This publication remains an indispensable reference book to all who are interested in American textile coloration and its related problems and also the constantly expanding range of American dyes.

H. Blackshaw

Veredler-Jahrbuch Deutscher Färberkalender 1956 (Volume 60)

Edited by O. Mecheels. Pp. iv + 368 + 58. Munich: Franz Eder Verlag. Price, $3.20.

The high standard which we have come to expect of this annual review of developments in dyeing and printing has been well maintained in the issue for 1956.

Once again the emphasis is naturally on the coloration of the newer synthetic fibres and of blends containing them, although newer methods of colouring the older fibres are not overlooked.

The achievement of 50 years of hydrosulphite production is marked by an interesting review of these products, and users of vat dyes will be interested in articles on the effect of modern washing agents on vat-dyed materials, on new levelling agents, and on printing with solubilised vat dyes.

The alphabetical list of new products, occupying some 55 pages and listing over 200 auxiliaries and dyes placed on the market by German and Swiss firms during 1955, testifies to the continued activity of these firms and adds materially to the usefulness of this most interesting and valuable publication.

A. THOMSON

Jahrbuch der Textilveredlung

Edited by G. Meier. Berlin: VEB Verlag Technik. Band 1: pp. 586; 1953; price, DM 25.00. Band 2: pp. 943; 1954; price, DM 36.00.

This yearbook is built up on much the same lines as the familiar and long established *Deutscher Färberkalender* in that it is a mélange of specialist articles, lists and particulars of new products made available in the year under review in each particular volume, and a survey of the year's literature. However, because of the larger size of the pages and the vast number of them, there is much more "meat" in this work than in the *Färberkalender*. The new dyes and textile auxiliary products, printing agents, etc. are dealt with by summarising the information given in their makers' literature. Unfortunately, only the products sold by the larger German manufacturers and merchants and the three largest Swiss dyemakers are dealt with. No reason is given for this partiality, but it does mean that the surveys do not by any means cover all, not even all the more important, new dyes, auxiliaries, and finishing agents which appeared during the years under review, for any such survey which ignores all mention of British, American, and French products, and those from the smaller German and Swiss makers, is bound to be inadequate from whatever point of view it is considered. On the other hand, the literature references—for they are only lists of books, papers, and patents—are much more complete, and appear to cover all the useful books and journals published in English. The articles which appear are written by a variety of authors. Not all of them are original, several being reprinted from other sources. They are rather uneven, both in length and in quality, some being little more than extended notes, others purely trade articles, others specialist articles, and yet others, e.g. the excellent account by K. Schäfer of pH in volume 1, being of the type expected in a textbook. Some, e.g. B. Hirsch's excellent account of the chemistry of textile auxiliary and finishing agents, appear in parts distributed between the two volumes. The articles cover the whole field of textile bleaching, dyeing, printing, and finishing. If the quality and the spread of the contributions can be maintained in succeeding years, then we shall have another standard work of reference to place on our shelves, providing that the editor realises—as he should from the literature references he has compiled—that dyes and textile auxiliary products of the highest standard, some indeed being important innovations, are produced in other countries besides Germany and Switzerland.

C. O. CLARK

The Journal of the Bradford Textile Society 1954–1955

Edited by G. G. Hopkinson. Pp. 130. Bradford. Price, 10s. 0d.

This annual publication fully maintains the high standards both of content and of production that it has set in previous years. Only one article, that on *Mélange Printing* by E. Isles, is of direct interest to the tinctorial technologist, but everyone connected with the wool industry in the West Riding will find the historical articles most absorbing; while indirectly several of the other articles, for instance *The Export Ambassador* by G. Phillips, are bound to be of interest to anyone engaged in the textile industry. C. O. CLARK

Proizvodstvo azokrasitelei [Manufacture of Azo Dyes]

By M. A. Chekalin and F. F. Eremin. 2nd edition 1952. Pp. 448. Moscow and Leningrad: Gosudarstvennoe nauchno-tekhnicheskoe izdatel'stvo khimicheskoĭ literatury. [In Russian.] Price, 8.75 roubles.

Although the dye industry provides much useful information, and even textbooks, on matters relating to the industries of its customers, it is understandably reluctant—being highly competitive in Western countries—to provide textbooks on the technological details of its own operations. In the U.S.S.R., however, such considerations can apply only when international competition must be considered, and it is therefore quite usual to see publications, such as the present one, which is essentially a book of "know how" for the man on the job and for trainees in the technical colleges. Only a very elementary knowledge of chemistry is assumed, though it is clearly intended that the reader will be making a collateral study of dye chemistry; the parts requiring a rather more advanced knowledge of chemistry are printed in smaller type and can be omitted in a first reading. Throughout the book, the hazards associated with the various operations are discussed, and the safety precautions to be taken are detailed. The text is profusely illustrated with process charts and diagrams of vessels, stirrers, pumps, filters, dryers,

grinding mills, etc., and even gas masks and the procedure for artificial respiration.

The contents, chapter by chapter, are briefly as follows— Raw materials and intermediates. General scheme of azo dye manufacture. Processes of diazotisation and coupling, as applied to intermediates of various types, and also methods of isolating the dye. Apparatus and machinery for all "wet" processes. Details of the manufacture of specific dyes (11 acid, 6 chrome, 18 direct, 2 oil-soluble, 1 disperse, and 3 lake-forming dyes, 3 lakes, and 5 other pigments). Processes of drying, grinding, and adjusting to standard strength, and the machinery used in these processes. Quality control and yield determination. Technical hazards and the precautions required. Labour organisation. Tables of solubilities, diazotisation conditions, coupling components, dyes, etc. are given as appendixes.

Each chapter, or section of a chapter, is followed by a group of questions for the student. There are 264 of these questions altogether, and from these the following examples are selected almost at random— What kinds of solutions can be used in rubber-lined apparatus, and to what temperatures may they be heated? What steps must be taken when plant is to be turned over from the manufacture of one dye to that of another? Explain the construction and operation of a vertical multi-chamber piston pump having ball valves. Name five possible causes of the production of unsatisfactory material in the manufacture of Acid Black S [*C.I.* 307]. Why is excessive dilution of the J acid solution harmful in the last coupling required in the manufacture of Direct Light-fast Blue [2-naphthylamine-4:8-disulphonic acid→1-naphthylamine→Cleve's acid→J acid (alkaline soln.)] and other similar azo dyes? What kinds of dryers are used for the drying of disperse dyes, and why cannot these dyes be dried in dryers of other types? Why must one not wash oneself with hot water after working with toxic materials?

A. E. STUBBS

Cellulose and Cellulose Derivatives
(High Polymers — Volume V)

Edited by E. Ott, H. M. Spurlin, and M. W. Grafflin. 2nd edition (in three parts). *Part I*: Pp. xvii + 1–509. 1954. Price, $12.00. *Part II*: Pp. ix + 511–1055. 1954. Price, $12.00. *Part III*: Pp. ix + 1057–1601. 1955. Price, $12.00.

This book is the second edition of a work that has been a standard reference text for cellulose chemists since its publication over twelve years ago. New viewpoints and developments in fundamental aspects have justified a complete revision of the first edition. The subject-matter has been brought up to date, within the limits imposed by the inevitable lag between writing and publication, and most of the chapters have been rewritten. Inclusion of new material and retention of much of the old have enlarged the book by 500 pages, and as a result it is divided into three parts, each bound separately, although pagination is continuous. It is a pity that author and subject indexes to the whole book appear only at the end of the third part. Considerable convenience to the reader would be gained if each part contained an index, even if a more comprehensive one were provided in the third part.

As before, the book is intended to describe the most important features of the chemistry and chemical technology of cellulose, but some consideration of physical phenomena is included. Each topic is contributed by a different American author, and their contributions have been so integrated that there is no serious overlapping. After an introductory chapter to Part I, the occurrence of cellulose is discussed, and there is a clear and logical survey of the structure and chemistry of cellulose. A chapter on structure and properties of cellulose fibres includes a very clear account of the interaction and arrangement of chains by H. Mark and sections on submicroscopic and microscopic structure and on structure–sorption relationships. Part I concludes with a chapter on the properties of woods and non-cellulosic materials associated with cellulose in Nature.

Part II is mainly concerned with the chemical technology of cellulose, and begins with a chapter on the preparation of cellulose from wood pulp, cotton, rags, and bast fibres. An up-to-date account of the bleaching and purification of wood cellulose is followed by another dealing with the properties and treatment of pulp for paper. The remainder, about two-thirds, of Part II is concerned with cellulose derivatives. A clear account of the chemical reactivity of cellulose is succeeded by a critical and comprehensive account of inorganic and organic esters, alkali and other metal derivatives, ethers, xanthates, and degradation. The preparation of viscose, the mechanism of xanthate and viscose formation, and the characteristics and reactions of viscose are discussed in detail. Some references to the dyeing and finishing of cellulose fibres are to be found in Part II, but the subject is not considered specifically or at any length.

Part III begins with a long chapter on solution properties of cellulose and its derivatives. There has been considerable fundamental work on this topic in recent years, and the sections on solubility, determination of molecular weights and their distribution, and theories of solution viscosity have been rewritten. There is a new section dealing with the properties of cellulose xanthate as a polyelectrolyte. Sections on thermodynamic and flow properties do not differ much from those in the first edition. Part II includes also a very good account of the mechanical properties of cellulose and its derivatives, much of which is applicable, in some degree, to fibres generally. There is a useful new chapter on tests for cellulose and its derivatives in which general methods, tests for raw materials, and chemical and physical tests are outlined and references, American and European, given for detailed standard methods. Part III concludes with an appendix giving such data as degree of

substitution and commercial viscosity relationships, qualitative data regarding solubility, a scheme for the identification of cellulose derivatives, trade and generic names, and some definitions and constants.

The book is very well documented with references and there are many clear diagrams. The three parts are well bound. Misprints or mistakes seem to be very few, and the book more than maintains the high standard of the first edition. It will be invaluable as a text and reference book for cellulose chemists generally and as a guide and stimulus to research. W. R. MOORE

Khimiya tsellyulozy i ee sputnikov [Chemistry of Cellulose and Associated Substances]

By Z. A. Rogovin and N. N. Shorygina. Pp. 679. Moscow and Leningrad: Gosudarstvennoe nauchno-tekhnicheskoe izdatel'stvo khimicheskoĭ literatury. 1953. [In Russian.] Price, 23 roubles.

There are some excellent books on cellulose in English, and this book in the Russian language, though an ably written treatise, is unlikely to be of much interest to English-speaking chemists. The treatment is based as far as possible on the Soviet classic on cellulose chemistry by the late Professor P. P. Shorygin (last edition, 1939); about five hundred pages are devoted to cellulose and its derivatives, about sixty pages to other polysaccharides associated with cellulose in Nature, and about one hundred pages to lignin. Most of the material can be found within the three volumes of "Ott" (see above), but the scope of the Soviet book is much narrower—technological matters are dealt with only briefly—and the emphasis is often different. For example, the chemical aspects of "weak places" in the cellulose chain and the associated question of the occurrence in the polymer chain of occasional straight-chain glucose residues and even residues of other monoses is allotted thirteen pages, corresponding to about one page in "Ott" (the work of Pacsu and its criticism by Reeves and others); but, on the other hand, the Soviet work contains nothing comparable to the full treatment given in "Ott" of the kinetics of the hydrolysis of cellulose and its relation to the possible occurrence of "weak places".

As would be expected, fields in which Soviet investigators have made notable contributions—for example, the nature of cuprammonium solutions of cellulose and the chemical changes produced by the oxidation of cellulose—are treated very fully.

A. E. STUBBS

Annual Reports on the Progress of Chemistry for 1954
Volume LI

London: The Chemical Society. 1955. Pp. 456. Price, 30s. 0d.

Following the usual pattern, the subject-matter of the present *Annual Reports* is distributed among six sections, each of which is appropriately subdivided to admit of special treatment of outstanding discoveries.

GENERAL AND PHYSICAL CHEMISTRY—The salient features of 1954 appear under the subheadings of *Molecular Spectra and Molecular Structure*, *Thermochemistry*, *Kinetics of Chemical Change*, and *Electrochemistry*. A useful additional service is the listing of some important general surveys and reviews which have appeared elsewhere during 1954, and these include papers on such subjects as the theory of the chemical bond, electron diffraction, microwave spectroscopy, and molecular structure studies by electron diffraction. Special attention is drawn in the report on *Thermochemistry* to the methods of combustion calorimetry, in which important developments are being made at the present time. There has continued a high level of activity in most fields of chemical kinetics and especially in the study of very rapid reactions—a class containing many examples of fundamental importance such as the combination of ions, and the reactions of atoms and simple molecules in the gas phase. The major contribution to the theory of chemical kinetics appears to be one of diffusion-controlled reactions presented by Noyes, wherein it is stressed that the instantaneous reactivity of a molecule that is produced in a reacting medium falls with the time which has elapsed since its formation, decreasing to a limit; several experimental tests are suggested. Continued interest is being shown also in the rates of chemical reactions near equilibrium. In the report on *Electrochemistry*, some aspects of recent work on aqueous solutions of electrolytes, and on the double layer and electrode reactions, are covered.

INORGANIC CHEMISTRY—Theoretical advances of considerable importance in the interpretation of the structures of many types of inorganic compound are recorded.

ORGANIC CHEMISTRY—Nearly one-third of the volume is devoted to this branch of chemistry. The theory of the effect of structure on reaction rate and equilibrium is intimately concerned with (*a*) the change of steric interaction effected by change in conformation in passing from reactants to transition state and from reactants to products respectively, and (*b*) the geometry of reactions; these factors are widely illustrated in the year's output of papers. Much work on hydrogen transfer is reported, the technique employed differing from the usual in that the hydrogen is supplied combined in a donor, usually *cyclo*hexene, instead of in the molecular form; it appears that palladium black and palladium–charcoal give excellent yields, whereas platinum catalysts are much less efficient, and Raney nickel is quite ineffective. By this method, successful reductions were obtained for a variety of aromatic nitro compounds, including nitrobenzonitriles and nitroacetophenones, but the nitroanilines, nitrobenzaldehydes, and *p*-dinitrobenzene did not react, while *m*-nitroaniline was obtained from *m*-dinitrobenzene. A new preparation of diazomethane, based on the alkaline decomposition of *N*-methyl-*N*-nitrosotoluene-*p*-sulphonamide, a stable and easily prepared material, is a welcome and serviceable discovery. Azobenzene formation from aniline and nitrosobenzene has been studied kinetically, and the

separation of the *cis* and *trans* forms by paper chromatography is described. As part of a more general study of hydrogen transfer, the kinetic dehydrogenation of hydroaromatic and other systems by quinones has been examined.

BIOCHEMISTRY— This field is so enormous that the reporters have limited their selection of topics to *Thermodynamic Data and their Application, The Chemical Nature of the "Active Centres" of Enzymes, Porphyrin Metabolism,* and *Biological Syntheses and Interconversions of some Monosaccharides.*

ANALYTICAL CHEMISTRY— The vast quantity of data in this field (555 references) are treated under such headings as *Reagents, Classical Organic Analysis, Instrumental Methods of Analysis, Chromatography, Electrophoresis, Ion Exchange, Flame Photometry,* and *Spectroscopic Methods.* Every chemist will be interested to learn that standard solutions of sulphuric acid can be prepared by means of specific-gravity determinations, since acid of concentration between 32% and 39% by weight is not hygroscopic and can be kept for at least six months without change; accordingly, standard solutions can be made by direct weighing of the aqueous acid between the above limits.

CRYSTALLOGRAPHY— Were it the intention of these *Reports* to be comprehensive, the available space could be filled merely by listing the references. Emphasis has therefore had to be restricted to structure analysis and to some of the results obtained thereby.

The value of this volume for all types of chemist may be realised from the facts that there are thirty pages of names in three columns in the author index and ten double-column pages of subject index, while the actual references total 3996, many of them composite.

H. H. HODGSON

Methoden der Organischen Chemie (Houben–Weyl)

Band IX

Schwefel-, Selen-, Tellur-Verbindungen

Edited by Eugen Müller. 4th edition 1955. Pp. xxxi + 1337. Stuttgart: Georg Thieme Verlag. Price, DM 218.00.

It has been found impossible to restrict the size of the present compilation of information on compounds containing sulphur, selenium, and tellurium on two grounds— (1) the outstanding importance of sulphur compounds in dye chemistry, for pharmaceutical preparations, for rodenticides and insecticides, for detergents, in the textile, rubber, and allied industries, and in biochemistry; and (2) because nothing precisely similar exists in the literature with respect to selenium and tellurium compounds, so that the present treatment amounts almost to a monograph on this now rapidly developing field of organic chemistry.

Accordingly, the volume is divided into two main parts as follows— Part I deals with the preparation and the reactions of sulphur compounds, and is subdivided into the 24 chapters on *Thiols and Thiophenols, Alkyl Hydrogen Polysulphides, Disulphides, Polysulphides, Sulphides (Thioethers), Ethylene Sulphides, Sulphonium Compounds, Mercaptals and Mercaptols, Sulphoxides and Sulphinimines, Sulphones and Sulphoximines, Sulphenic Acids and their Derivatives, Aliphatic Sulphinic Acids, Aromatic Sulphinic Acids, Aliphatic Sulphonic Acids and their Derivatives, Sulphochlorination by the C. F. Reed Process, Aromatic Sulphonic Acids, Aromatic Sulphonic Acid Anhydrides, Aromatic Sulphonyl Halides, Functional N-Derivatives of the Arylsulphonic Acids, Aromatic Sulphonic Esters, Thiosulphonic and Thiosulphinic Esters, Thioaldehydes and Thioketones, Thio Acids and their Derivatives,* and *Sulphur-containing Derivatives of Carbonic Acid.* Part II gives similar information on selenium and tellurium compounds. A short chapter is included on *The Nomenclature of the Sulphur Compounds.*

The treatment of the sulphur compounds is based on the different valencies exhibited by sulphur, beginning with the lowest, and the chapter on sulphochlorination is included because of the importance of the Reed process. A large separate chapter is given to the aromatic sulphonamides in view of their practical importance in the dye industry, for synthetic tanning materials, for softening agents, for mothproofing chemicals, and in chemotherapy.

The literature references are complete to 1954, while as many as possible have been included for 1955, and publications from other countries are impartially cited. The comprehensive character of this huge volume may be realised from the fact that the author index occupies 58 pages of names in three columns and the subject index 63 double-column pages.

For over 40 years organic chemists have been accustomed to consult "Houben–Weyl" for advice on the most suitable reagents and conditions for effecting a given reaction or employing special apparatus, and the present volume brings this service right up to date for sulphur, selenium, and tellurium compounds. This volume is a worthy addition to the series and will receive a warm welcome from all organic chemists.

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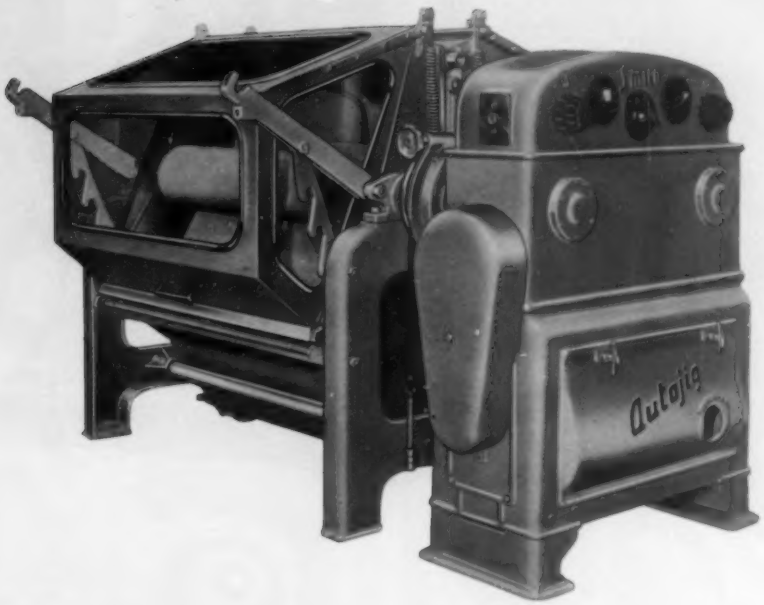

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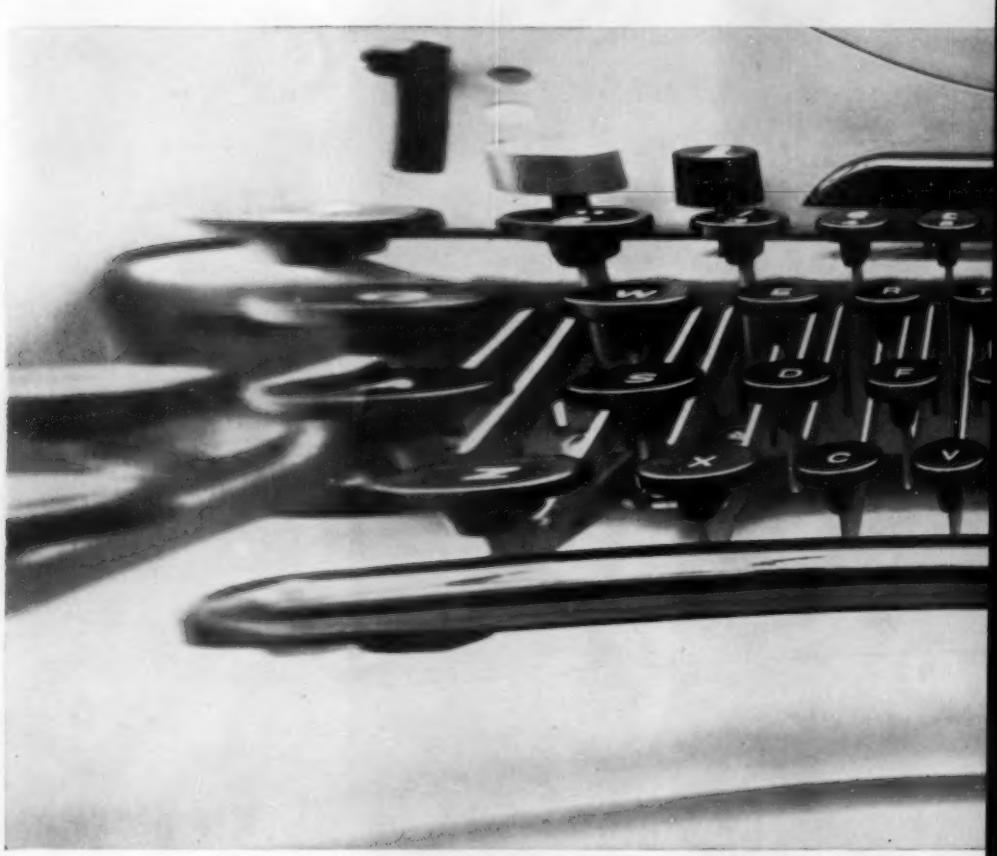

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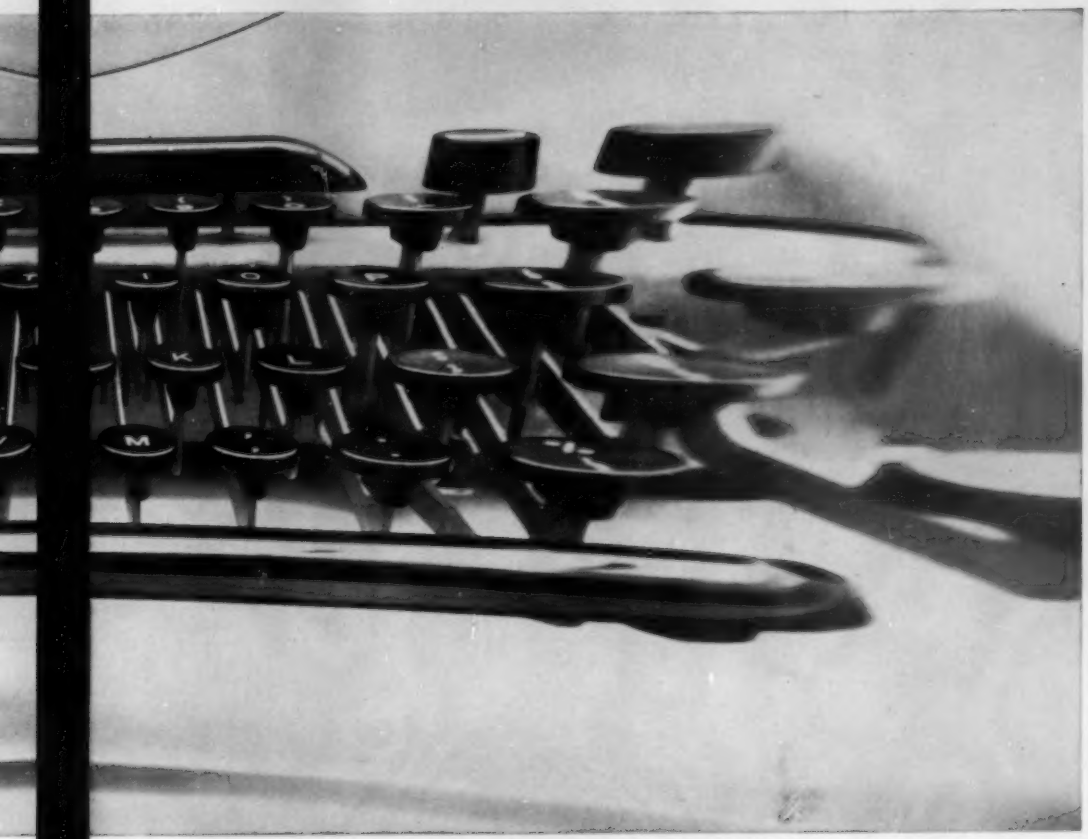

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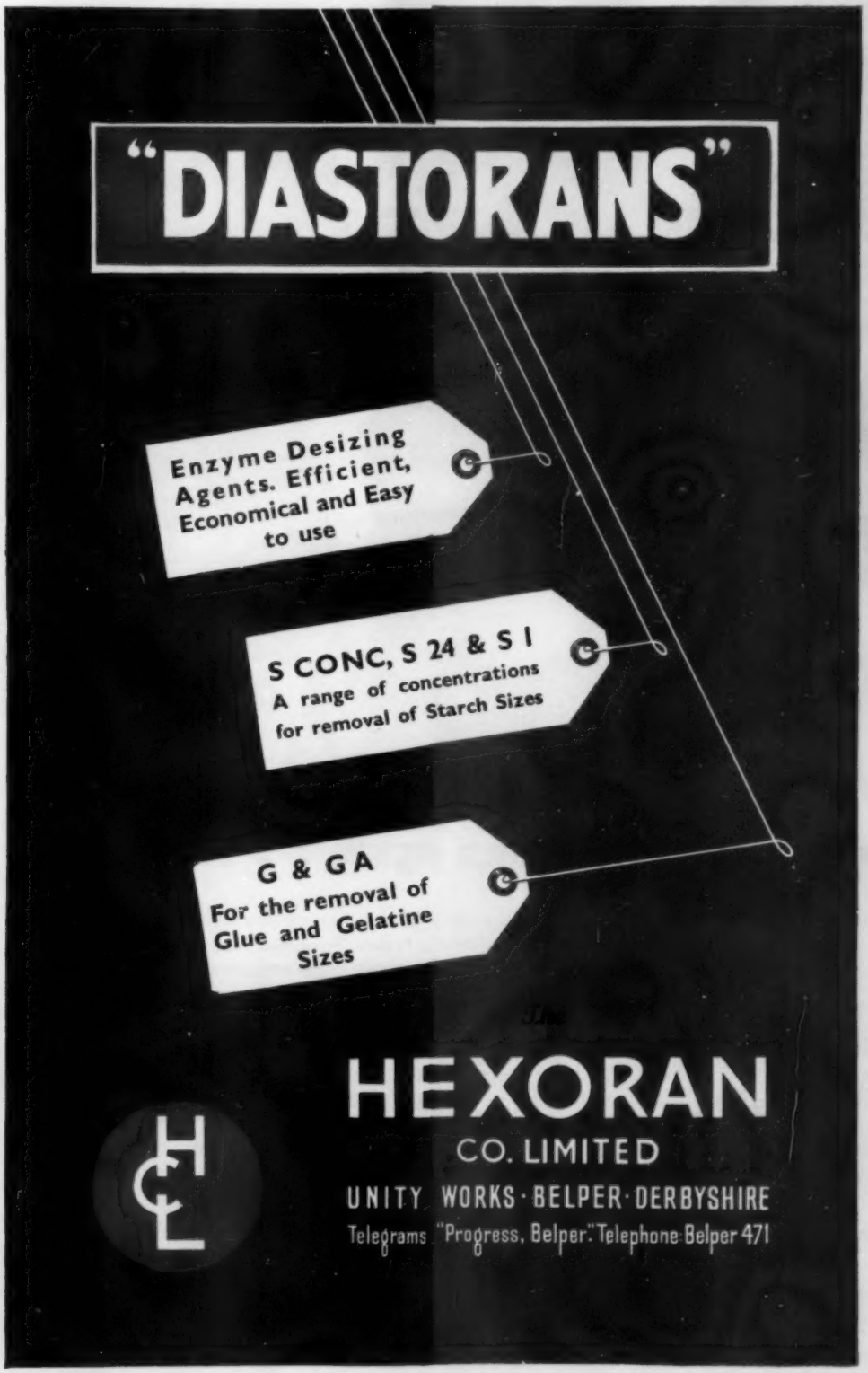
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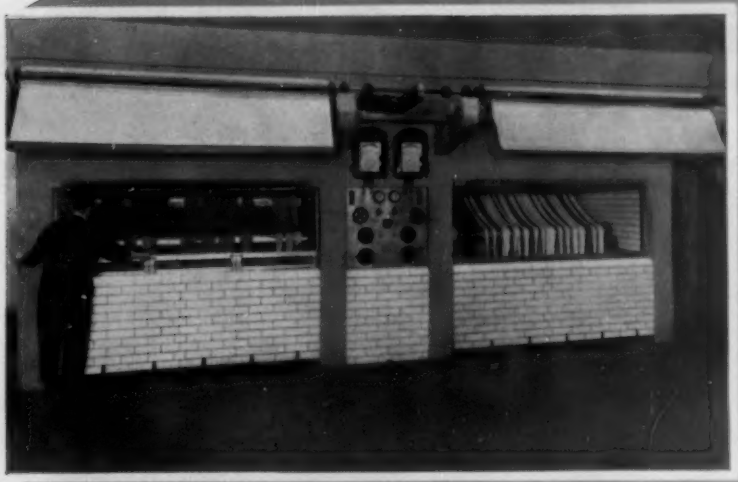

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*Trade Mark

DU PONT Dyes

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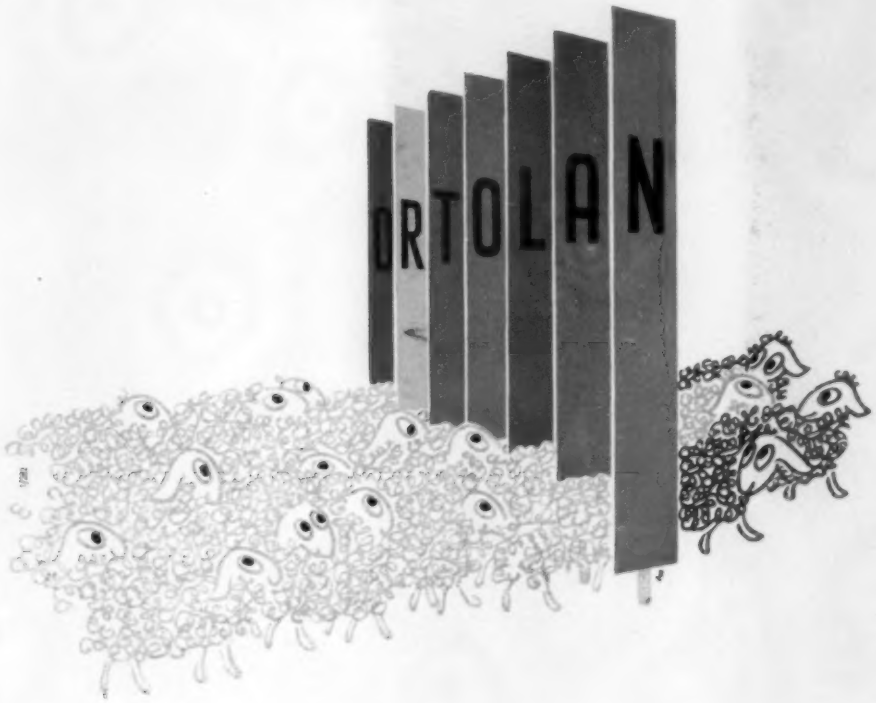

Wool!

This dirty, greasy fibre in its raw state emerges after numerous treatments as a highly attractive material in countless beautiful shades – thanks to the skill and ingenuity of the textile processor . . . and the colour chemist.

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ORTOLAN DYESTUFFS

The Ortolans, suitable for dyeing wool in practically all forms, possess many outstanding characteristics:–

satisfactory fibre protection under weakly acid dyeing conditions / short dyeing time / excellent fastness / very good levelling properties.

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SANDOZ

Phosphoric Acid

The use of Phosphoric Acid offers many technical advantages in the dyeing and processing of wool, e.g.

Improved handle and less damage to wool on prolonged boiling, compared with sulphuric acid.

Brighter and faster shades, due to elimination of any iron reaction.

Considerable saving in time with more level results in the dyeing of fast to milling acid colours and chrome colours.

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SOLE DISTRIBUTORS OF THE PRODUCT TO THE DYEING AND TEXTILE INDUSTRY UNDER BRITISH PATENT No. 588,454 FOR THE MANUFACTURERS

ALBRIGHT & WILSON LIMITED
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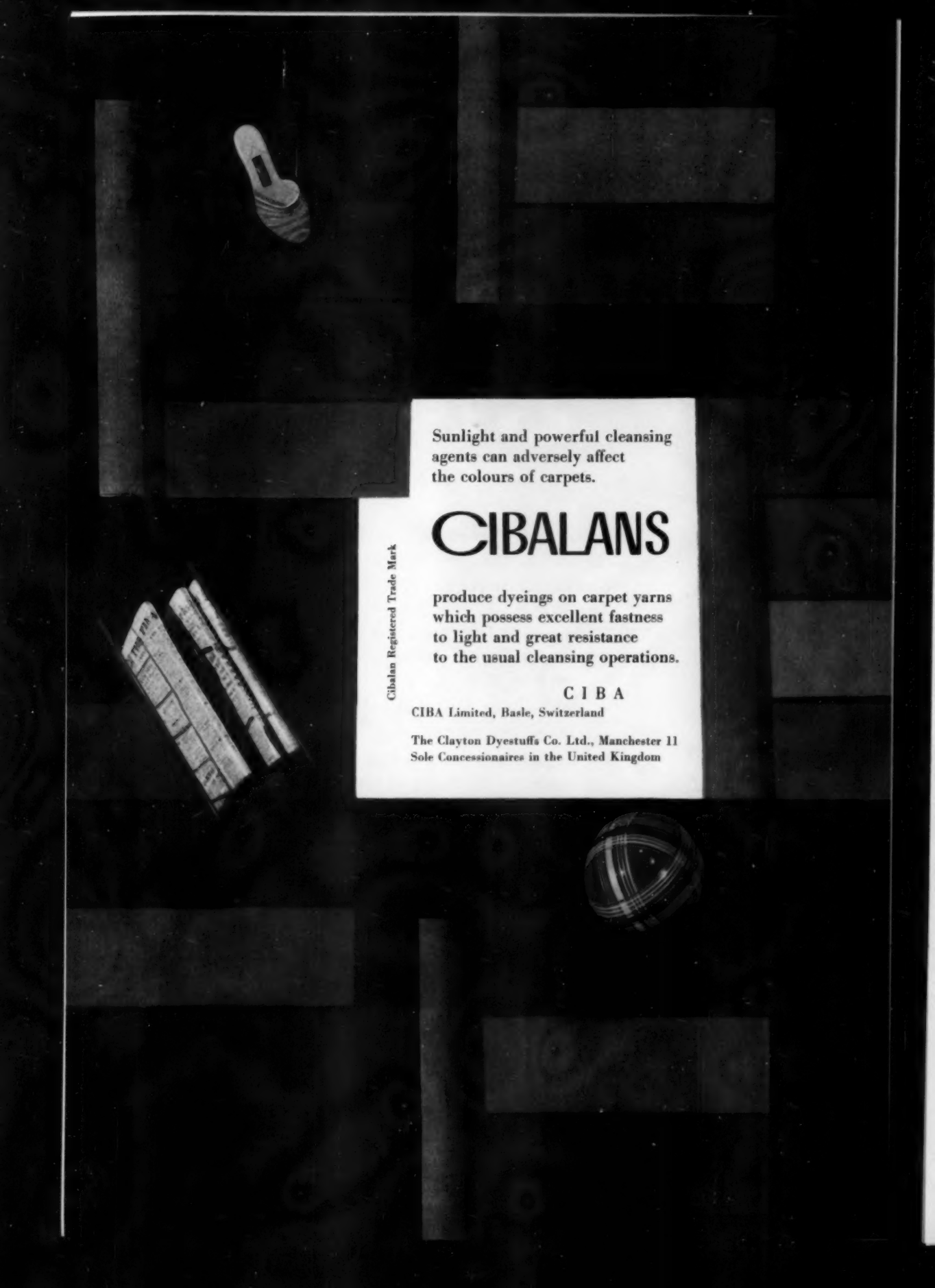
Sunlight and powerful cleansing agents can adversely affect the colours of carpets.
CIBALANS
produce dyeings on carpet yarns which possess excellent fastness to light and great resistance to the usual cleansing operations.
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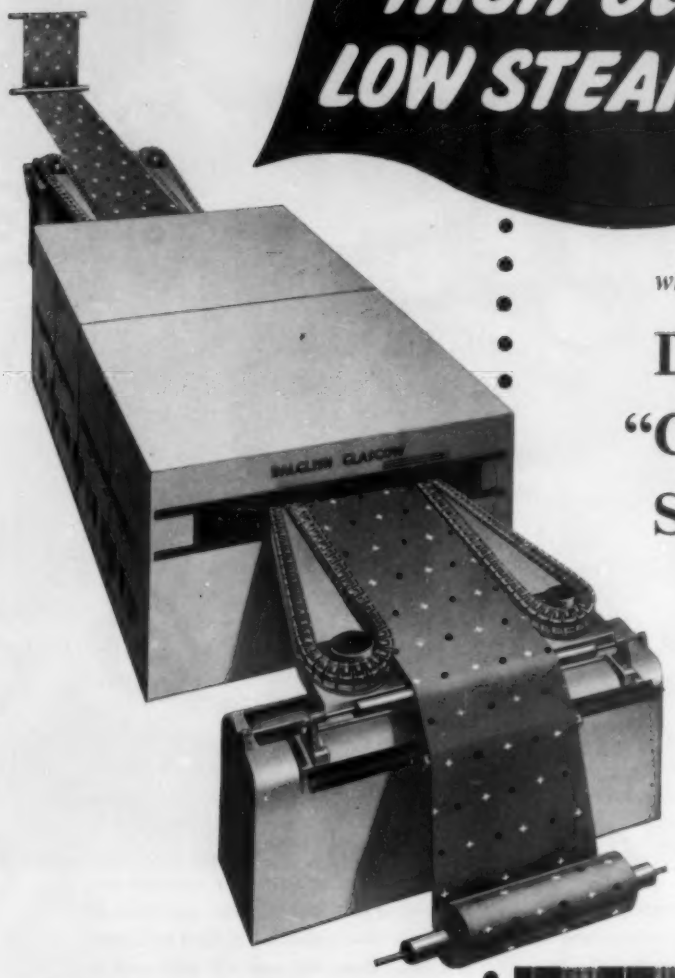

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Dalglish "Comasach" Stenter

Exceptionally high evaporation, with resultant increased rate of drying, makes the DALGLISH "COMASACH" the most economic and efficient stenter now available . . . as an example — 100 yards of 5 oz. cotton per minute in a compact 50 foot chamber — and steam costs cut by 50% ! ! When supplied as a pin, or pin and clip stenter, the DALGLISH "COMASACH" is fitted with patent DALGLISH electronic guiders and positive overfeed. Whatever your cloth — natural or synthetic, woven or knitted — specify "COMASACH". Write now for the "COMASACH" illustrated brochure giving performance figures and other details.

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No. 5 *'The Swan' Lavenham, Suffolk*

(With acknowledgement to Trust Houses, Limited)

In Lavenham High Street, at the junction of three highways from London, Ipswich and Bury St. Edmunds, stands the Swan a 14th century coaching inn at one time the focal point of all Suffolk.

The name Lavenham means wool-town or wool-village. Wool so enriched and empowered the merchants of Lavenham that the town was once rated 12th in importance in all England.

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The Swan offered rest and refreshment to men and beasts. It was also the workrooms of a rich clothier. And many a load was passed straight to the weavers' gallery overlooking the courtyard.

Wool, Britain's staple trade for centuries, is still one of our greatest national assets. Although now it has settled into a less picturesque background of mechanical looms and mechanised transport.

Continuous laboratory research too, enables us to produce great quantities of competitively-priced cloth, dyed with good and fast colours and specially processed to meet an increasing number of specific demands. This very important scientific work is conducted by firms like Brotherton.

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One of the world's largest manufacturers of hydrosulphites, liquid sulphur dioxide and hexamine.
Makers of an extensive range of Metachrome dyes for dyeing wool in all its forms.
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A 36

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LOOSE WOOL and SLUBBING

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* Economical * High light- and wet-fastness properties

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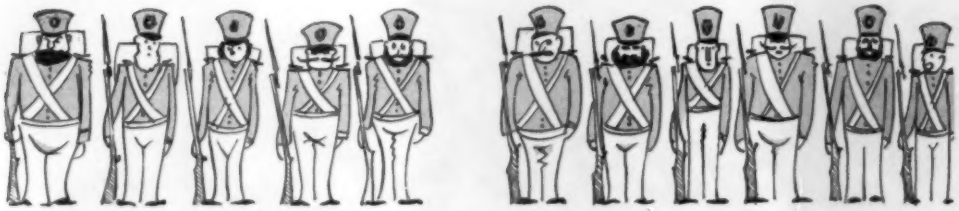
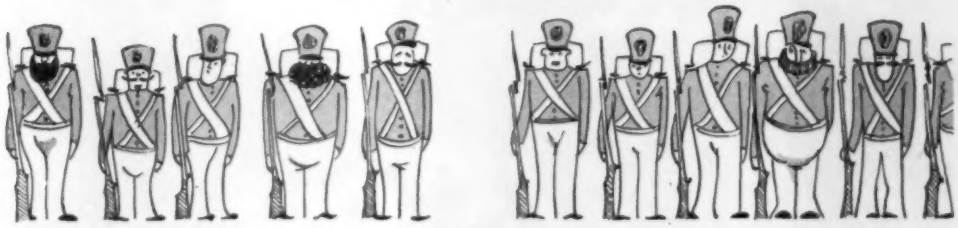
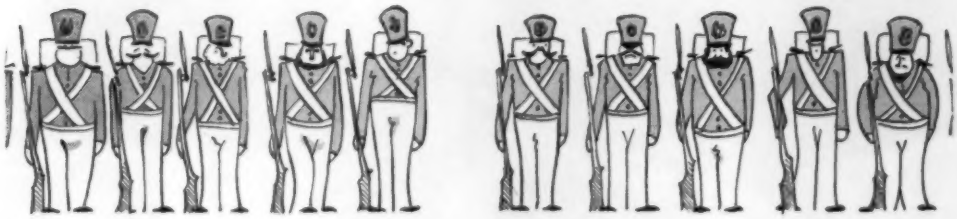


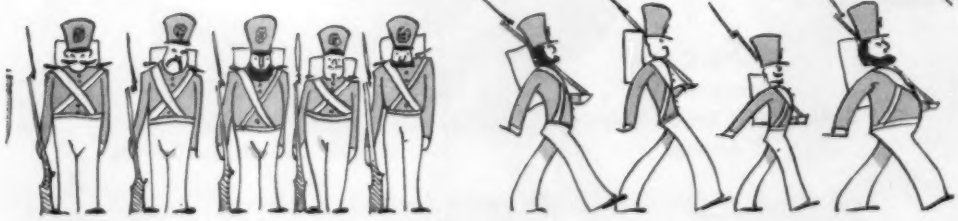
Nervanase & Gelatase



on parade for sizing



de-sizing and finishing



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BENZANIL FAST

Bright red shade with good fastness to light and wet processing.

Excellent for dyeing viscose rayon in yarn, cake and piece.

Suitable for crease resist.

Reserves acetate.

YDC

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The Sign of Good Colour

Town End Chemical Works Limited

BRAMLEY • LEEDS

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University

Compagnie Française des Matières Colorantes

THE DYEING AND PRINTING OF NYLON— This booklet (Technical Notice No. 55E) contains collected information on the dyeing of nylon and nylon–cotton and nylon–wool mixtures, and also on direct and discharge printing with disperse (Nyloquinone), acid, neutral-dyeing metal-complex (Inochrome), and direct cotton dyes. Notes on setting, scouring, desizing, bleaching, and antistatic and anti-snag finishing are included, and full details of the fastness characteristics of the dyeings are given.

The Geigy Co. Ltd.

ERIOCHROME BLACK SR— This chrome black may be dyed afterchrome or on a chrome mordant on all forms of wool materials, but it is particularly suitable for dyeing high-quality yarn and cloth, since it is the most level-dyeing of the chrome blacks and can be applied from a sulphuric acid dyebath to leave cellulosic effect threads undyed. Fastness figures include— Light 6, washing (S.D.C. Test 2) 4–5, perspiration (S.D.C. Test) 3–4, severe milling 2–3.

IRGALAN BRILLIANT GREEN 3GL— This neutral-dyeing metal-complex dye is recommended for use alone and in mixtures with other Irgalan and Irganol S dyes for greens on all types of wool goods, and on silk and polyamide fibres, on all of which it gives dyeings having the excellent fastness properties characteristic of this range of dyes. Fastness figures on wool include— Light 6–7, washing (*b*) 4–5, heavy milling 4–5.

IRGALAN DARK BROWN 5R— This card contains dyeings on wool cloth of five mixtures of Irgalan dyes in four depths giving a range of browns based on the Dark Brown 5R, which is recommended for such colours. It is also suitable for browns which are required to redden in artificial light.

SOLOPHENYL RUBINE 3BL— This direct cotton dye gives bright reddish violets of very good fastness to light on cellulosic fibres. The S.D.C. classification is A, and it gives solid dyeings on viscose rayon of non-uniform affinity and on cotton–viscose rayon mixture materials. Aftertreatment with Tinofix A or LW improves the wet fastness. Acetate rayon is reserved white. Fastness figures on viscose rayon include— Light 7–8, washing (*a*) 3, perspiration 2–3.

Imperial Chemical Industries Ltd.

BRENTOGEN BLUE B— This is a homogeneous, stabilised azoic combination giving bright medium blues on cellulosic materials. It is applied in the normal manner with caustic soda and is developed by acid steaming. Alternatively, it may be developed by treatment in an acid bath without steaming if a slight loss in yield may be tolerated. It may be developed in neutral steam using Brentogen Developer N. Fastness figures of a print on cotton at standard depth for Brenthols include— Light 4–5, washing (repeated mild, 5 times at 40°C.) 5, hypochlorite ("dry chemicking") 4–5.

CALEDON BLUE 3RC— This vat dye gives reddish blues on cotton, linen, and viscose rayon and is also of interest on silk where good fastness to degumming is desired. On cellulosic fibres maximum colour value is obtained by Method 2 at 50°C., but since it is virtually insensitive to over-reduction at high temperatures, showing no significant deterioration in tinctorial value, hue, or fastness when vatted and dyed at 80–90°C., advantage may be taken of the improvement in levelling properties with rise in dyeing temperature. All brands are suitable for conventional leuco-dyeing processes. The FD and Paste Fine brands are recommended for pigmentation processes and the Standfast molten-metal process. Fastness figures for a standard-depth dyeing on cotton include— Light 6–7, washing (Test No. 5, 5 times at 100°C.) 4–5, soda boil–hypochlorite bleaching 4.

CALEDON BRILLIANT RED 5B— This homogeneous vat dye gives bright bluish reds on all types of cellulosic material. Method 2 at 50°C. is recommended, or alternatively Methods 1 and 3. The leuco compound is stable at high temperatures (ca. 90°C.), and advantage may thus be taken of the higher migrating power at such temperatures to obtain level, well penetrated dyeings. This is particularly important, as Dispersol VL is of little value as a levelling agent for this dye. All brands are suitable for leuco-vat dyeing, while the Paste Fine and FD Powder Fine brands should be used for pigmentation processes. The colour on viscose rayon is much yellower than on cotton. This dye has no affinity for silk and is of little interest in printing. Fastness figures on cotton include— Light 6–7, washing (repeated severe, 5 times at 100°C.) 4–5, soda boiling–hypochlorite bleaching 4.

DURAZOL SKY BLUE G— This homogeneous direct dye gives bright greenish blues of good fastness to light on cotton and very good fastness to light on viscose rayon. The S.D.C. classification is A. This dye is recommended for dress goods, ribbons, etc. where brightness of hue and fastness to light are important rather than high wet fastness. Crease-resist finishing only slightly reduces the light fastness provided that the usual alkaline soaping is included. It covers viscose rayon of irregular dyeing affinity and is of interest for solid dyeings on cotton–viscose rayon mixtures. Fixanol PN aftertreatment of dyed cotton reddens the hue but appreciably improves the washing fastness. The GP brand is recommended for direct printing cotton, viscose rayon, silk, wool, and chlorinated wool. Fastness figures on cotton include— Light 5, washing (Test No. 1, once at 40°C.) 2, perspiration 1.

ICYL SKY BLUE FL— This further addition to the range of dyes giving level dyeings on viscose rayon of irregular affinity gives blues of brightness and light fastness hitherto unobtainable with dyes of this class. It is suitable for dyeing materials subsequently to be crease-resist-finished. Fixanol PN aftertreatment considerably increases the fastness of dyeings to water, but their washing fastness is not improved, and they become very much redder. Fastness figures on viscose rayon include— Light 6, washing (Test No. 1, once at 40°C.) 2, water 3.

TECHNICAL INFORMATION LEAFLETS— *Dyehouse No. 272. The Colouring of Polyester Resin–Glass Fibre Laminates*— The stability has been assessed of a range of pigments in a typical polyester resin, Marco SB 28C, applied to glass cloth to form a laminate by the wet lay-up technique and cured. Colour instability, pigment flocculation, and effect on curing properties have been noted, and the daylight fastness of the coloured laminates has been determined.

Dyehouse No. 274. The Dyeing of Cellulose Triacetate Fibres— Supplement No. 2. The Production of Black Shades— Three methods of producing azoic blacks are described. No. 1 is preferred, in which the base (Dispersol Diazo Black B or 2B) is applied first, Brentosyn BB second, and then diazotisation and coupling. Method 2 gives a more intense black by applying Dispersol Diazo Black A first, diazotising cold, and coupling with Brentosyn BB in a third bath; but, although dyeings of good light fastness are obtained, their fastness to heat-setting and steam-pleating is poor. Method 3 is analogous to that for producing azoic dyeings on acetate rayon, nylon, or Terylene, in which base and coupling component are applied together. Dyeings of good fastness to heat-setting and pleating are obtained, but this method is unsuitable for jig-dyeing below 100°C. and does not give full blacks on staple fibre below 100°C. It is thus restricted to piece dyeing where high-temperature (> 100°C.) machinery is available and for dyeing bright continuous-filament yarn at 100°C. and spun yarn above 100°C.

Dyehouse No. 275. White Discharges on Durazol Blue 8G Grounds— A recipe is offered which has given reasonably satisfactory white discharges on cotton and viscose rayon under laboratory conditions.

Dyehouse No. 276. Resist Salt L: Printing on Nylon— An addition of Resist Salt L to the paste when printing Dispersol, Duranol, and Solacet dyes has been recommended to prevent reduction of dye in the print paste and during steaming. It has been shown that residual Resist Salt L, which cannot be removed by washing, causes yellowing of the pattern on exposure to light and an apparent decrease in the light fastness of the dye. It is now recommended that Resist Salt L should not be used when printing nylon except, possibly, with very heavy prints. Sodium chlorate is suggested as a possible alternative, but its suitability should be determined with each dye combination.

Dyehouse No. 277. Vat Dyestuffs on Woolled Sheepskins— A special technique for dyeing woolskins with selected vat dyes is described. Dyeing is continued for 30 min. at 50°C., and the skins are then hydroextracted and oxidised at 50°C. for 20 min. in a solution of potassium persulphate and acetic acid.

Dyehouse No. 279. Alcian Dyestuffs: Dyeing of Cellulosic Fibres— The value of the various Alcian X dyes for dyeing purposes has been assessed, and recommended methods of application are described. Alcian Blue 8GX 300 can be applied to a variety of cellulosic materials to give brilliant turquoise blues hitherto unobtainable with the same standard of fastness. Greens 2GX and 3BX are of less value. Little success has been obtained with mixtures of Blue 8GX and Yellow GX. The latter may be used for golden yellows. Dyeing processes described include winch and jig dyeing, continuous dyeing, and hank and package dyeing. Fastness data on cotton, linen, and viscose rayon are included.

Dyehouse No. 283. The Dyeing of Terylene Polyester Fibre: Supplement No. 6. The Application of Selected Vat Dyestuffs in Pigment Form— Certain vat dyes in pigment form are absorbed by Terylene at elevated temperatures (130°C.) in a similar manner to disperse dyes. Dyeings so produced are characterised by extremely high fastness to all wet treatments, steaming processes, and dry heat, much superior to anything obtainable with disperse dyes. Four dyes are particularly recommended—FD Caledon Golden Yellow GK Powder Fine, Durindone Scarlet YS Paste, Durindone Red BS Paste, and Caledon Brilliant Violet R Powder Fine. Dyeing is started at 70°C., the temperature raised slowly to 130°C., and dyeing continued at 130°C. for 60 min. A "reduction-clear" is recommended, except in the case of pale dyeings, to remove dye particles deposited on the surface of the fibres, which would result in poor fastness to rubbing and wet treatments.

Dyehouse No. 285. The Effect of the Specific Gravity of the Printing Paste on Effective Formosul Concentration— Starch–tragacanth and British gum vat thickenings may have sp.gr. of 1·25–1·3 and 1·35–1·4 respectively. The effect these relatively high values have on the effective Formosul concentration is not always appreciated, and figures are given for converting concentration calculated on a wt./wt. basis to the corresponding wt./vol. basis.

Dyehouse No. 286. The Use of V-brand Pigment Pastes in Leather Finishing— The V-brand pigments can be used in exactly the same way as the Neran Brilliant dyes to produce bright finishes having better fastness to light and rubbing than is obtainable with the latter. They are suitable also for aniline-type finishes.

Resins No. 6. The Effect of Thickeners and Extenders in Water Paints based on Paralac 10E.

M.C.S.D. No. 7. The Surface Dressing of Roadways—Adhevia ACL, An Alternative to Adhevia T.

Vat, Sulphur, and Acid Milling Dyestuffs on Cotton–Nylon Mixtures— This card contains six dyeings on cotton–nylon drill containing 17% of nylon. In three cases the cotton component has been dyed with vat dyes and sulphur dyes have been used for the remainder. The nylon has been dyed with neutral-dyeing acid dyes. In those cases where the depth of colour required allows, the nylon has been dyed during the soaping-off of the vat dyes with Lissapol N. When sulphur dyes are used and with heavy dyeings it is better to dye the nylon separately after soaping. Although many vat dyes are absorbed to some extent by the nylon component and their fastness to light on nylon is low, since the polyamide fibre content is low and the fibres are intimately blended, it has been possible to treat the material as 100% cotton with the recommended vat dyes. The sulphur (Thionol) and soluble sulphur (Thionol M) dyes can be applied by the method normally used for dyeing 100% cotton, and without exception leave the polyamide fibre virtually undyed.

Azoic and other Fast Dyeings on Knitted Cotton Fabric— Reprint of a lecture by E. R. Wiltshire (J.S.D.C., 70, 41 (Feb. 1954)).

The Dyeing of Mixtures of Terylene Polyester Fibre and Wool— Reprint of a lecture by A. S. Fern and H. R. Hadfield (J.S.D.C., 71, 277 (June 1955)).

The Dyeing of Terylene Polyester Fibre with Disperse Dyes above 100°C.— Reprint of a paper by A. S. Fern (J.S.D.C., 71, 502 (Sept. 1955)).

The Dyeing of Textile Fibres above 100°C.— Reprint of a lecture by J. A. Fowler (J.S.D.C., 71, 443 (Aug. 1955)).

An Explanation of Dyeing Mechanisms in Terms of Non-polar Bonding— Reprint of a lecture by A. N. Derbyshire and R. H. Peters (J.S.D.C., 71, 530 (Sept. 1955)).

Some Observations on the Dyeing of Wool at High Temperatures— Reprint of a lecture by G. A. Coutie, D. R. Lemin, and H. Sagar (J.S.D.C., 71, 433 (Aug. 1955)).

Hexagon Digest No. 22— Articles in this issue include *Aqueous Dispersions of Resins in Water Paints* by W. Peace and G. D. Rack, *Colorimetry and Colour Gamuts* by T. Vickerstaff, *The Generation and Dissipation of Static Electricity in the Textile Industry* by J. Earnshaw, and *The Vibroscope* by M. M. Cross and G. Grimshaw.

Sandoz Products Ltd.

Artisil Direct Orange GFL— This disperse dye gives bright yellowish oranges on acetate rayon, cellulose triacetate, and synthetic fibres. Dyeings on these fibres have very good fastness to light, wet treatments, sublimation, and gas fumes. Tone-in-tone dyeings are obtained on acetate rayon–nylon mixtures. It is particularly suitable for use alone and in mixtures on polyester fibres and on polyacrylonitrile fibres when dyed above 100°C. Dispersions fine enough for package dyeing are readily obtained. Fastness figures on acetate rayon include— Light (daylight) 7, washing (SNV 60°C.) 4–5, gas fumes 5.

Synopsis of Dyestuffs and Chemicals for the Paper Industry— Dyes, grouped according to their class, are recommended for specific uses, and chemicals are suggested for particular purposes.

Lanasyn Bordeaux RL; Dark Violet RL; Yellow GLN; Yellow 3GL— Dyeings on wool of each of these neutral-dyeing metal-complex dyes, used alone and as components of two-component mixtures, are given, together with fastness data for dyeings on wool and nylon, on single cards issued as additions to Pattern Card No. 1223/53. Fastness figures on wool include—*Bordeaux RL:* Light (daylight) 7, alkaline milling (strong) 5; *Dark Violet RL:* Light 6–7, alkaline milling (strong) 5; *Yellow GLN:* Light 7, alkaline milling (strong) 5; *Yellow 3GL:* Light 6–7, alkaline milling (strong) 4–5.

Lanasyn Bordeaux RL; Yellow GLN; Yellow 3GL— Dyeings of each of these dyes on wool and nylon sliver are mounted on separate cards.

Lanasyn Bordeaux RL and Dark Violet RL on Unweighted Silk; Lanasyn Yellow 3GL on Unweighted Silk— Dyeings of these dyes in two depths on cloth and as a component of a mixture dyeing on yarn are included together with fastness data in these two additions to Pattern Cards No. 1220/54 and 1220/55. Fastness figures include— *Bordeaux RL:* Light (daylight) 7, washing (60°C.) 3–4; *Dark Violet RL:* Light 6, washing (60°C.) 4–5; *Yellow GL:* Light 7, washing (60°C.) 4.

Solar Orange 4GN— This Class B direct dye gives bright oranges on cellulosic fibres. Dyeings have very good fastness to light, and are unaffected by crease-resist finishing. It is particularly recommended as a basis for fast-to-light browns. Type AC8 leaves acetate rayon white. Fastness figures include— Light 6, washing 3, hot pressing (after 2 hr.) 4–5.

Abstracts from British and Foreign Journals and Patents

The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952), and also, together with symbols and the periodicals abstracted, in the annual index*

I—PLANT; MACHINERY; BUILDINGS

PATENTS

Continuous Blending of Pigments in Spinning Dopes
Celanese Corpn. of America *BP* 743,625
Apparatus is described for continuously feeding at a fixed rate a spinning solution and a liquid concentrate containing the pigment. W.G.C.

Generation and Use of Acoustic Energy to Accelerate Wet Treatment of Materials
F. Masse *USP* 2,702,260
Rapid cleansing or dyeing is obtained by setting up sound waves in the treating liquor in such a manner that any standing wave phenomena which might prevent portions of the materials from being exposed to high-intensity sound are eliminated. There is relative motion between the sound wave pattern and the position in the liquid of the materials being treated. C.O.C.

Transmitting Vibrations to Liquors through the Walls of the Container
Thermofrigor *BP* 742,366
In a vessel having at least four sides two or more electro-magnetic vibrators act on different walls of the vessel. The vibrations are arranged so as to be energised in phase with one another. C.O.C.

Winch Dyeing Machine
V. C. Bohm *USP* 2,702,464
Satisfactory dyeing is obtained below the boil and with the material travelling at $\frac{1}{20}$–$\frac{1}{15}$ the normal speed if a continuous stream of dye liquor intermingled with air (or other non-soluble gas) impinges upon the material during all or most of the dyeing time. This enables delicate materials and/or colours to be dyed in rope form. C.O.C.

Perforated Beam for Treating Fabrics with Gas or Liquid
Burlington Engineering Co. *USP* 2,701,957
A perforated beam on which fabric is wound to be treated with gas or liquid, of simple design, giving minimum weight with maximum strength, is described. C.O.C.

Ager
Tower Iron Works *BP* 742,207
An ager in which dripping is prevented, the conveyor rails do not touch the printed surface, and an oxygen-free atmosphere is maintained. C.O.C.

Treating Animal Skins and Similar Pervious Sheet Materials with Liquid
Secotan *USP* 2,702,229
The skin is placed upon a porous bed. An impervious membrane of thin flexible, preferably somewhat elastic, material is laid over the entire bed and a fluid pressure differential is applied across the bed and membrane to force the membrane against the bed. The treating liquid is then introduced between the skin and the membrane under pressure and at such a rate that it covers the skin but does not separate the membrane from the parts of the bed not covered by the skin. During introduction of the treating liquid the fluid pressure is maintained in the membrane. C.O.C.

Automatic Washing, Drying, or Dry-cleaning Machine
Avco Manufacturing Co. *BP* 742,264

Machine for Ironing Garments or Fabrics
S. G. Widdowson *BP* 742,816

Paper-coating Machines
Paper Patents Co. *BP* 741,970
The coating machine, applying e.g. mineral pigments to high-quality paper, includes a main coating roll supported on a pivotally mounted balanced frame and an auxiliary train of rolls on a supplemented framework mounted pivotally by the main frame. This arrangement ensures that the force applied to auxiliary rolls and the nip pressure of the main rolls can be finely adjusted, only small pressure is required to engage or disengage the rolls, the pressures between the rolls as well as the movement of the web during coating can be more easily controlled. K.W.

Surface Finishing of Hat Bodies, etc.
M. Casso *BP* 743,344
Mechanism for pumicing, sanding, polishing, friezing, etc. hat bodies or the like. C.O.C.

Spray Gun
S. G. Dehn *BP* 735,983
A conventional spray gun is fitted with an auxiliary jet set at right angles to the main jet so that catalyst or curing agent may be injected into and mixed with the sprayed coating material. E.C.

Technical Developments in Pigments and Paints during 1955 (V p. 118)
Continuously Depositing Gelatin in or on Travelling Strip Material (XI p. 127)
Conditioning Machine for Fur Coats or the like (XII p. 128)

II—WATER AND EFFLUENTS

Split-treatment Softening of Water
J. R. Roesum
Ind. Eng. Chem., **47**, 2313–2317 (Nov. 1955)
Split-treatment softening of water often offers considerable economic advantages over conventional treatment. Equations are derived which make it possible, from an analysis of the raw water, to estimate the results to be expected from split-treatment softening. The lime, lime–soda, and lime–zeolite processes are considered. By means of the equations, the proportion of water to be by-passed, and the chemical dosages needed to produce an effluent of the desired quality, can be calculated. W.K.R.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Sulphonation with Sulphur Trioxide—Anhydride Formation with Detergent Alkylate
E. E. Gilbert and B. Veldhuis
Ind. Eng. Chem., **47**, 2300–2303 (Nov. 1955)
The material responsible for the development of acidity in aqueous slurries of sodium dodecylbenzenesulphonate prepared by using sulphur trioxide vapour as the sulphonating agent has been isolated and identified as the sulphonic acid anhydride. The most practical way of destroying the anhydride is addition of 1% water to the crude sulphonic acid immediately after sulphonation, which causes rapid and quantitative hydrolysis to sulphonic acid. Destruction of the anhydride also stabilises the colour of the product. W.K.R.

Effect of Micellar Size on Physicochemical Properties of Surface-active Agents
A. M. Mankowich
Ind. Eng. Chem., **47**, 2175–2181 (Oct. 1955)
The physicochemical properties of 1% solutions of an anionic and a non-ionic detergent were compared at different micellar sizes, obtained by adding different builders. It is shown that micellar solubilisation, carbon suspensibility, spreading coefficient, and interfacial tension are functions of micellar size. A more fundamental unit of solubilisation, No. of molecules of dye (Orange OT) solubilised per micelle, was developed. W.K.R.

Stability of Dilute Alkaline Solutions of Hydrogen Peroxide
W. D. Nicoll and A. F. Smith
Ind. Eng. Chem., **47**, 2548–2554 (Dec. 1955)
A study of the decomposition of dilute solutions of hydrogen peroxide over the pH range 10–12·5 has shown that the tendency to decompose more rapidly with increase in alkalinity is not due to the increase in hydroxyl ion concentration, but to increased catalytic activity of small

amounts of certain cations (e.g. of Cu, Fe, etc.) present. Relatively stable solutions can be obtained within this pH range by forming soluble or insoluble complexes with the cations by addition of e.g. dimethylglyoxime or sodium silicate, or by their physical removal. The mechanism of stabiliser action is discussed. The catalytic activities of different cations are specific, but subject to modification by concentration, pH, and other cations or anions.

W.K.R.

Preparation of an *N*-Hydroxymethyl Derivative of Steelon (6-Nylon) soluble in Methanol and Ethanol

S. Chrzczonowicz, L. Brajter, and A. Nowakowski

Zesz. Nauk. Politech. Łódz., No. 9 (*Chemia* 3), 63–72 (1955)

A 92% yield of an *N*-hydroxymethyl derivative of Steelon was obtained by treating 10 g. of polymer dissolved in 40 g. of formic acid (90%), warming up to 60°C., and adding during 30 min. 10 g. of paraformaldehyde suspended in 18 g. of water. Steelon was found to be depolymerised by the experimental conditions to the extent of 29%. The formaldehyde derivative thus produced is very elastic and transparent. It can be used for imparting a matt appearance and a rough surface to steelon. A method for determining formaldehyde chemically combined with steelon is described; it was found to be 2·38%. Attempts to produce this derivative using formalin were not successful.

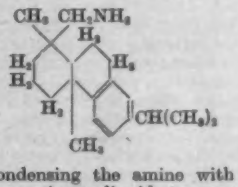
T.Z.W.

PATENTS

Surface-active Compounds

General Aniline *USP* 2,703,797

The *N*-polyethenoxy derivatives of dehydroabietinyl primary amine, which amine is of formula—



obtained by condensing the amine with 4–40 mol. of ethylene oxide, are viscous liquids to waxy solids, soluble in water and having surface-active properties. Those containing 8–30 ethylene oxide radicals per mol. of amine are excellent detergents, especially for cellulose.

C.O.C.

Detergents from *N*-Monoalkylglucamines

Commercial Solvents Corpn. *USP* 2,703,798

The products obtained by condensing an *N*-monoalkylglucamine with an aliphatic ester of a fatty acid are excellent detergents which need no purification. In their manufacture only the one reaction occurs

$$R^1NH\cdot CH_2\cdot(CHOH)_4\cdot CH_2OH + R^2COOR^3 \rightarrow R^3OH + R^2\cdot CO\cdot NR^1\cdot CH_2\cdot(CHOH)_4\cdot CH_2OH.$$

C.O.C.

Quaternary Ammonium Compounds— Detergents, Emulsifying Agents, and Textile Assistants

American Cyanamid Co. *USP* 2,703,795

Compounds of formula

$[R^1\cdot CO\cdot NH\cdot C_nH_{2n}\cdot NR^2R^3\cdot CH_2\cdot CHOH\cdot CH_2\cdot O\cdot CO\cdot R^4]^+X^-$

(R^1 = aliphatic radical of > 7 C; R^2 and R^3 = CH_3 or C_2H_5; R^4 = hydrocarbon radical of a rosin acid; n = 2–10; X = Cl, Br or I), e.g. the product obtained by heating 3-dimethylaminopropylamine with tall-oil fatty acid at 120–130°C. and treating the resulting condensate with tall-oil rosin acid and an epihalohydrin at > 200°C., are surface active and useful as detergents, textile assistants, etc.

C.O.C.

Cleaning Compositions comprising Anionic Surface-active Agents

Scottish Oils *BP* 741,478

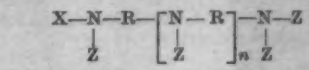
Useful detergent, dry cleaning and spotting agents comprise metal (usually Na) alkyl sulphates preferably obtained by sulphating a distillation fraction of shale oil (range 180–330°C., preferably 200–330°C.), together with water, a mineral oil, e.g. white spirit fractions, and an alcohol or ether of high mol. wt., e.g. *cyclo*hexanol or diethylene glycol alkyl ethers. A single liquid phase is possible under wider ranges of relative proportions, and the amount of alcohol or ether required to give a single-phase product is reduced by adding an NH_3 or metal salt, e.g. ammonium sulphate. In one example a spotting agent comprises Na alkyl sulphate (20% aq. soln.) containing up to 5% Na_2SO_4 (52%), water (15), kerosene b.p. 165–275°C. (25), $(NH_4)_2SO_4$ (5), and dimethyl*cyclo*hexane (3).

J.W.B.

N-Carboxymethyl-substituted Alkylene and Polyalkylene Polyamines— Simultaneous Sequestering and Surface-active Agents

Monsanto *BP* 742,055

Materials having simultaneous surface-active and alkaline earth metal-ion sequestering properties comprise a substituted alkylene or polyalkylene polyamine of formula—



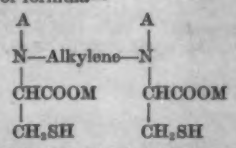
(X = keryl group ("keryl group" refers to a mixture of branch-chain alkyl groups) derived from monochlorokerosene; R = alkylene group of 2 or 3 C; n = 0, 1 or 2; Z = H or a group of formula $-CH_2\cdot COOM$ (M = H, alkali metal, or NH_4), at least one Z being $-CH_2\cdot COOM$).

J.W.B.

Dimercaptoalkylenediaminepolycarboxylic Acids— Chelating Agents

Dow Chemical Co. *BP* 742,996

Compounds of formula—



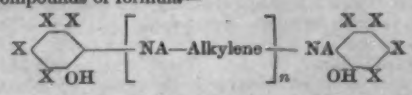
(Alkylene = CH_2CH_2, $CH(CH_3)\cdot CH_2$ or $CH_2CH_2CH_2$; A = H or CH_2COOM; M = H, NH_4, K, Na or an amine), e.g. disulphhydrylethylenediaminetetracarboxylic [*sic*] acid, are very powerful water-soluble chelating agents for alkaline earth metals and for Fe, Co, Ni, Mn and Zn.

C.O.C.

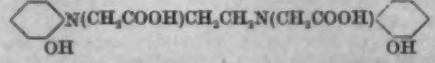
Hydroxy Aromatic Alkylenepolyaminopolycarboxylic Acids— Chelating Agents having Bactericidal and Fungicidal Properties

Dow Chemical Co. *BP* 743,126

Compounds of formula—



(Alkylene = CH_2CH_2, $CH_2CH_2CH_2$ or $CH(CH_3)CH_2$; n = 1–5; X = H, OH, Hal, Alk or alkoxyl; A = CH_2COOM or CH_2CH_2COOM; M = H, alkali metal, ammonium or subst. ammonium), e.g.



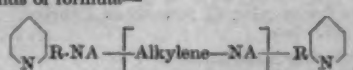
form extremely stable metal complexes. The free acids and their chelates are soluble in organic solvents and oils. The alkali metal or ammonium salts are soluble in water but their chelates are less soluble in neutral solution and they can be used to precipitate metal ions as chelates, in alkali however the chelates are unexpectedly soluble probably because of secondary salt formation either as hydrates or phenolates or both.

C.O.C.

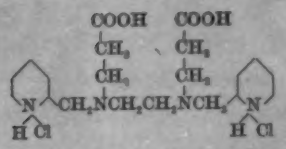
Dipyridyl Aliphatic Alkylenepolyaminepolycarboxylic Acids— Chelating Agents

Dow Chemical Co. *BP* 743,683

Compounds of formula—



(A = acyl; R = a direct bond, methylene, ethylidene or a group containing a chain of $<$ 13 C the C atoms of the chain being saturated with H atoms or alkyl radicals containing fewer C atoms than the chain; Alkylene includes *cyclo*alkylene and interposes 2 or 3 C between the N atoms; n = a whole number) and their acid-addition salts, e.g.



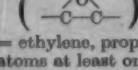
are soluble in water as are their metal chelates. As the chain length of R increases the solubility of the compounds and their metal chelates diminishes but at the same time their emulsifying and surface active properties increase. Increase in n increases solubility in water and chelating power for heavy metals. C.O.C.

Epoxy Aliphatic Polyaminoacetic Acids—Chelating Agents
Dow Chemical Co. *BP* 742,945

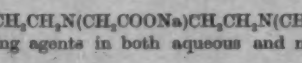
Compounds of formula—

$$R(NH\text{-}Alkylene)_nNH_2$$

(R = an aliphatic hydrocarbon group containing several C atoms to two adjacent C atoms of which there is attached an O atom



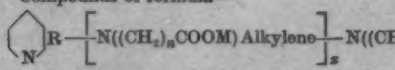
n = 1–3; Alkylene = ethylene, propylene or trimethylene; of all the amino H atoms at least one of those in the NH_2 group has been displaced by CH_2COOM; M = H, alkali metal, NH_4 or subst. NH_4), e.g.



are chelating agents in both aqueous and non-aqueous solutions. C.O.C.

Pyridyl Aliphatic Alkylenepolyaminepolycarboxylic Acids—Chelating Agents
Dow Chemical Co. *BP* 742,441

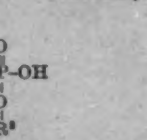
Compounds of formula—



(R = chain of < 13 C saturated with H or monovalent alkyl radicals containing fewer C atoms than the chain; Alkylene includes *cyclo*alkylene and interposes 2 or 3 C atoms between the N atoms; n = 1 or 2; x = 1–4; M = H, Na, K, Li, Ce, Rb, NH_4 or a substituted ammonium group) are powerful sequestering agents for alkaline earth and rare earth metals and other metal ions, e.g. Pb, V, Zr and U which are difficult to chelate. They may be made e.g. from a chloroalkylpyridine and an alkylene polyamine by the process described in *BP* 726,268 (*J.S.D.C.*, **71**, 255 (May 1955)). C.O.C.

Mono-acid Phosphoric Esters—Emulsifying Agents
ICI *BP* 743,752

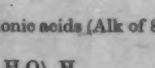
Compounds of formula—



(R^1 = Alk of < 5 C; R^2 = a branched dodecylphenyl radical of the alkylphenol formed by alkylating phenol with the propylene tetramer of boiling range 181–218°c.) are liquids completely miscible with hydrocarbons. They are used as emulsifying agents for oil-in-water emulsions of mineral oils and of glyceride oils. Such emulsions do not rust ferrous metals. C.O.C.

Emulsifying Agents
Atlas Powder Co. *BP* 742,626

The salts of alkylarylsulphonic acids (Alk of 8–16 C) with compounds of formula—



(R = hydrocarbon of 12–20 C; $m + n$ = 3–30) are emulsifying agents which yield stable emulsions in presence of calcium and/or magnesium. C.O.C.

Starch Ethers
Corn Products Refining Co. *BP* 741,742

Starch is etherified by treating it at 20–55°c. with an alkylene oxide, e.g. ethylene oxide, in presence of an alkaline etherification catalyst, e.g. 0·025–0·075 mol. of NaOH per anhydroglucose unit, together with a salt which will react with the alkylene oxide in presence of water to liberate free OH ions and produce additional alkaline etherification catalyst, e.g. 6% by wt. (on starch) of NaCl where NaOH is used as the catalyst. The products have improved properties, including negligible gelling and retrogradation on standing. J.W.B.

Calcium Salts of Nitrogen-containing Polymers—Sizes
Monsanto *BP* 743,606

Products useful as sizing and stiffening agents for textiles are obtained by treating polyacrylonitrile with a suspension of calcium hydroxide until the calcium salt has a nitrogen content of 3·5–5·5%. W.G.C.

Amine–Formaldehyde Resin
J. Wolf & Co. *BP* 736,955

Dicyandiamide (1 mol.) is condensed with formalin (3·6–5·0 mol.) at 95°c. and pH > 7 for 4 hr. The solution is spray-dried to a stable resin which may be used in aqueous solution with an acidic catalyst to impregnate fibrous materials, e.g. leather or fabrics, and is cured by heating at 120–250°c. Fabrics thus treated show better crease-resistance than is obtained with conventional treatment. E.C.

Rot- and Insect-proofing Agents
E. B. Higgins and A. E. Read *BP* 742,782

Compounds of formula—

$$X\cdot O\cdot PO(OR^1)\cdot OR^2$$

(R^1 = pentachlorinated phenyl; R^2 = subst. or unsubst. phenyl; X = H or acyl) e.g. the product obtained by treating pentachlorophenol at 200–300°c. with phosphorus oxychloride, are excellent rot- and insect-proofing agents for application to textiles. They can be applied in acid or neutral dyebaths. C.O.C.

Adhesives for Cellulose Acetate and Bonding of Weftless Ribbons (X p. 125)
Synthetic Resinous Coatings for rendering Fabrics and Leather Water-impermeable but Vapour-permeable (X p. 126)
Influence of Oxidisability of Oil-containing Substances on Rayon Materials. III—Analytical Methods (XIV p. 131)

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Reaction of Sulphonation. XXXVII—Equilibrium between a Sulphonic Acid and its Acid Chloride
A. A. Sprŷskov and N. V. Apar'eva
J. Gen. Chem. U.S.S.R., **25**, (12), 2286–2288 (Nov. 1955)

Sodium 2:5-dichlorobenzenesulphonate is dissolved in a mixture of chlorosulphonic and sulphuric acids, and the equilibrium attained in the reaction—

$$2R\cdot SO_2\cdot ONa + Cl\cdot SO_2\cdot OH + H_2SO_4 \rightleftharpoons R\cdot SO_2\cdot Cl + R\cdot SO_2\cdot OH + 2NaHSO_4$$

is examined at 20°c. The equilibrium constant for this reaction is found to be 0·33. Equilibrium is established most rapidly when the chlorosulphonic acid content of the original mixture is high. A.E.S.

Preparation of *o*- and *p*-Acetamidobenzaldehydes
J. J. Brown and R. K. Brown
Canadian J. Chem., **33**, 1819–1823 (Dec. 1955)

Colour-forming Characteristics of Benzyl Cyanide Couplers
M. Kusakabe and I. Yakame
Repts. Govt. Chem. Ind. Research Inst., Tokyo, **50**, 172–178 (1955)
Chem. Abs., **49**, 15578 (25 Nov. 1955)

Thirteen $C_6H_3CH_2CN$ derivatives were adhered step-wedge-wise on standard movie positive films, colour-developed after 10 sec. exposure to artificial sunlight, and then tested for their chromogenic properties (solubility in the developer, affinity for the oxidised developer, and spectral transmission of developed films). The following six (all containing NO_2) proved to be excellent magenta (unless otherwise stated) couplers: 4-, 2- and 3-$NO_2C_6H_4CH_2CN$ with λ_{max} 515, 500 (red orange) and 510

(light magenta) mμ. respectively and 4-, 5- or 6-NO_2-3-$BrC_6H_3CH_2CN$ λ_{max} 510, 510 and 505 (orange) respectively. C.O.C.

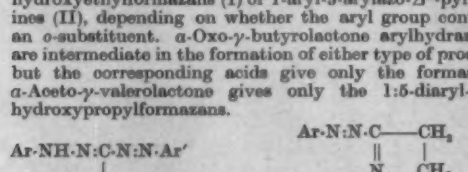
Absorption Spectra of Dyes in Solution
Y. Oshika.
J. Phys. Soc. Japan, **9**, 594–602 (1954)
Chem. Abs., **49**, 15483 (25 Nov. 1955)
Theoretical. C.O.C.

Primary Products of the Interaction of Amines with Nitrous Acid
B. I. Stepanov, E. A. Vinnikov, and E. S. Lisitsyna
J. Gen. Chem. U.S.S.R., **25**, (9), 1794–1798 (Sept. 1955)

Earl and co-workers (*J.C.S.*, 1937–1939) suggested the formation of a cpd. containing the grouping $\rangle N{\cdot}N(OH)_2$ (formed by direct addition of nitrous acid to an amine) as the primary product in diazotisation and nitrosation, and Grachev (*J. Gen. Chem. U.S.S.R.*, **18**, 848 (1948)) considered that this substance could be identified with the unstable "amine–nitrite" obtained by treating an amine salt with sodium nitrite soln. at very low temp. "Amine–nitrites" are now prepared from aniline, *o*-, *m*-, and *p*-toluidines, 1-naphthylamine, *N*-ethylaniline, and *NN*-diethylaniline. They are found to react with diazomethane with formation of the free amine and methyl nitrite; *N*-ethyl-*N*-nitrosoaniline and *NN*-diethyl-*p*-nitrosoaniline do not react with diazomethane. These facts support the view that "amine–nitrites" are simply salts formed by the amine with nitrous acid. In particular, the fact that a tertiary amine, diethylaniline, can form an "amine–nitrite" indicates that such substances cannot be dihydroxy compounds of the type postulated by Earl. A.E.S.

Reaction of Diazonium Salts with α-Aceto-γ-lactones
G. F. Duffin and J. D. Kendall
J.C.S., 3470–3474 (Oct. 1955)

The reactions of benzenediazonium salts with α-aceto-γ-butyrolactone in neutral or weakly alkaline soln. are known to give the pale yellow α-oxo-γ-butyrolactone phenylhydrazone, but in strongly alkaline soln. aromatic diazonium salts are now found to yield 1:5-diaryl-3-2′-hydroxyethylformazans (I) or 1-aryl-3-arylazo-Δ^2-pyrazolines (II), depending on whether the aryl group contains an *o*-substituent. α-Oxo-γ-butyrolactone arylhydrazones are intermediate in the formation of either type of product, but the corresponding acids give only the formazans. α-Aceto-γ-valerolactone gives only the 1:5-diaryl-3-2′-hydroxypropylformazans.



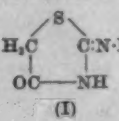
(I) (II)
H.H.H.

Relation of Physical Properties of Carcinogens to their Activity. I—Basicity of Alkylated 1-Aminoazobenzene Dyes
E. Sawicki and F. E. Ray
J. Org. Chem., **19**, 1686–1692 (1954)
Chem. Abs., **49**, 16766 (25 Nov. 1955)

An account of the spectrophotometric measurements of the basicity of a series of 4-aminoazobenzene dyes in 50% ethyl alcohol. The change in basicity caused by substitution of various alkyl groups in the molecule is explained on the basis of electronic theory. C.O.C.

Preparation of 5-phenylazo-2-arylimino-4-thiazolidones and the Corresponding 5-Amino Compounds
B. K. Patnaik and M. K. Rout
J. Indian Chem. Soc., **32**, 563–565 (Aug. 1955)

2-Arylimino-4-thiazolidones (I) have been coupled with diazotised aniline to give the corresponding 5-phenylazo compounds. These on reduction with sodium hydrosulphite give 5-amino-2-arylimino-4-thiazolidones.



(I)

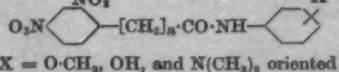
(R = phenyl, *p*-tolyl, *m*- and *p*-chlorophenyl, *m*- and *p*-nitrophenyl, *p*-carboxyphenyl, and α- and β-naphthyl). C.H.R.

Separated Chromophoric Systems
XXIX—Comparative Spectroscopic Investigations on 4-Nitrobenzylidene and 4-Nitrobenzyl Derivatives of Aromatic Amines
V. A. Izmail'skii and E. A. Smirnov
J. Gen. Chem. U.S.S.R., **25**, (7), 1400–1412 (July 1955)

The colours—judged visually and measured spectroscopically—of pairs of compounds, $R^1{\cdot}C_6H_4{\cdot}CH{:}N{\cdot}C_6H_4{\cdot}R^2$ (I) and $R^1{\cdot}C_6H_4{\cdot}CH_2{\cdot}NH{\cdot}C_6H_4{\cdot}R^2$ (II) (R^1 = H, NO_2; R^2 = H, CH_3, OH, $O{\cdot}CH_3$), are compared in the dissolved state (by transmission) and in the form of powders (by reflection). When $R^1 = NO_2$, II is more deeply coloured than I in the solid state, whereas, in accordance with generally accepted views on the effect of the length of the conjugated system, I is more deeply coloured than II in solution. These results are to be explained by the occurrence of intramolecular action between the two distinct separated chromophoric systems in II, which can occur to an appreciable extent only when the molecules are close together, as in the solid state. This intramolecular effect is greater in II because the electron-donating effect of benzylamino is greater than that of benzylideneamino. A fuller exposition of these views will be found in earlier papers (see J.S.D.C., **67**, 198 (1951); **68**, 464 (1952); **71**, 622 (Oct. 1955)). The views of Hodgson on the colour of such compounds (J.S.D.C., **64**, 184 (1948)), which is an interpretation of the results of Ufimtsev (*J. Gen. Chem. U.S.S.R.*, **11**, 846 (1941)), which are based on the occurrence of σ,π-conjugation (hypereonjugation), are considered to be groundless. Hodgson states, incorrectly, that *N*-4-nitrobenzylaniline is colourless [Hodgson gives Ufimtsev as the source of this information], and this assumption lies at the basis of his argument; moreover, he applies his theory to two compounds, which are represented to be those investigated by Ufimtsev, but are assigned incorrect structural formulae [the abstract of Ufimtsev's paper given in *Chem. Abs.*, **36**, 4110 (1942) indicates that the compounds concerned have 4-nitrobenzylamino groups attached to benzene nuclei in acenaphthene and fluorene, respectively, whereas Hodgson represents them as substituted in methylene groups]. A.E.S.

XXX—Colour Phenomena in Arylamides of 4-(2:4-Dinitrophenyl)butyric Acid
E. A. Smirnov
Ibid., (12), 2336–2343 (Nov. 1955)

The work on colour due to external interaction of separated chromophoric systems in 4-*p*-nitrophenylbutyric arylamides (see J.S.D.C., **71**, 622 (Oct. 1955)) is now extended to the corresponding 2:4-dinitrophenyl compounds—



(*n* = 3; X = $O{\cdot}CH_3$, OH, and $N(CH_3)_2$ oriented *meta* and *para*), and an analogous comparison is made with compounds for which *n* = 1 and 2 (investigated previously—*J. Gen. Chem. U.S.S.R.*, **17**, 309 (1947)). In the main, the results are similar to those obtained for the mononitro compounds. A.E.S.

Absorption Spectra and Molecular Structure
A. A. Kharkharov
IV—Effect of the Character of the Substituent on Colour in Substituted Phenylhydrazones of Acridine-9-aldehyde Hydrochloride
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 880–889 (Sept.–Oct. 1955)

Work on the absorption spectra of soln. of arylhydrazones of acridine-9-aldehyde in alcoholic HCl is continued (see J.S.D.C., **71**, 472 (Aug. 1955)). *para*-Substituted phenylhydrazones are used, the substituents in order of increasing λ_{max} (given in parentheses in mμ.) being—NO_2 (522), CHO (539), $CO{\cdot}CH_3$ (540), $SO_2{\cdot}NH_2$ (540), COOH (546), NH_2}Cl (550), Cl (554), $SO_2{\cdot}OH$ (557), Br (560), –NH– (dihydrazone with 2 mol. of aldehyde) (560), H (562), SCN (565), I (566), $CH{:}N{\cdot}NH{\cdot}C_6H_5$ (570), $N{:}N{\cdot}C_6H_5$ (579), CH_2 (580), $N{:}N{\cdot}C_6H_4{\cdot}O{\cdot}CH_3$ (590), $NH{\cdot}CO{\cdot}C_2H_5$ (596), $NH{\cdot}CO{\cdot}CH_3$ (598), OH (610), $O{\cdot}CH_3$ (610), $O{\cdot}C_2H_5$ (610), $NH{\cdot}C_6H_5$ (655), $NH{\cdot}C_6H_4{\cdot}O{\cdot}CH_3$ (715). The results

demonstrate the hypsochromic and bathochromic effects of electron-accepting and electron-donating substituents respectively, and also bathochromic effects due to the lengthening of the conjugated chain.

V— Spectroscopic Investigation of Solutions of Chlorine-substituted Phenylhydrazones of Acridine-9-aldehyde Hydrochloride in Alcoholic Hydrogen Chloride

Ibid., 1071–1077 (Nov.–Dec.)

The investigation is extended further to the *o* (533), *m* (548), 2:4 (523), 2:5 (510), 3:4 (540), 2:4:5 (510), 3:4:5 (525), 2:3:4:6 (509), and 2:3:5:6 (512) chlorine-substituted phenylhydrazones of acridine-9-aldehyde hydrochloride (max. of principal absorption band is given in parentheses in mμ.). The hypsochromic effect of chlorine increases in the order *para* < *meta* < *ortho*, in accordance with the increase in inductive effect. The effect of the accumulation of substituents is very roughly additive. The positions of the ultraviolet maxima vary very little among these cpd.

A.E.S.

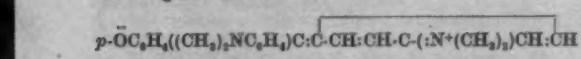
Colour and Constitution. I— Dyes with Crossed Absorption Systems

S. Hünig, H. Schweeberg, and H. Schwartz

Annalen, **587**, 132–145 (1954)

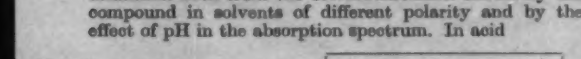
Chem. Abs., **49**, 15820 (25 Nov. 1955)

The dyes α,α-di(*p*-dimethylaminophenyl)quinone methide (I) and α,α-di(*p*-dimethylaminophenyl)anthraquinone methide (II) can be considered to have the two absorbing structures



(Ia)

The relative contributions of structures I and Ia can be estimated both from the solvatochromism shown by the compound in solvents of different polarity and by the effect of pH in the absorption spectrum. In acid



(III)

is formed. Comparison of the spectra of I and II with those of α-phenyl-α-(*p*-dimethylaminophenyl)-quinone methide (IV) and α-phenyl-α(*p*-dimethylaminophenyl)anthraquinone methide (V) and study of their changes in various solvents and at different pH, revealed that the spectra of I and II showed contributions from both the Malachite Green type of absorption and of the dimethylaminofuchsin chromophore, although the Malachite Green type of structure is less important in both II and V. Preparation of IV and V is described.

C.O.C.

Spectrographic Investigation of a Unimolecularly Spread Dye Film

H. Käufer and G. Scheibe

Z. Elektrochem., **59**, 584–587 (1955)

Chem. Abs., **49**, 15483 (25 Nov. 1955)

The characteristic monomeric absorption band was not detected in unimolecular films of 5:6:5′:6′-dibenzopseudoisocyanine on water, even when the light was passed many times through the film by means of mirrors. The characteristic polymeric absorption band was seen in thicker films formed by compressing the unimolecular film by a force of ≮ 12 dynes/cm. Presence of this band is reversible.

C.O.C.

Cyanine Dyes. VI— 1-*m*-Nitrophenylquinaldinium Perchlorate and its Reactions

G. T. Pilyugin and Z. Ya. Krainer

J. Gen. Chem. U.S.S.R., **25**, (12), 2271–2274 (Nov. 1955)

The title cpd. is prepared for the first time by the acid condensation of *m*-nitrodiphenylamine with paraldehyde, and it is converted by conventional methods into four cyanine dyes, viz.— bis[1-*m*-nitrophenyl-2-quinoline]trimethincyanine, [1-*m*-nitrophenyl-2-quinoline][1:3:3-trimethyl-2-indolenine]trimethincyanine, [1-*m*-nitrophenyl-2-quinoline][3-ethyl-2-benzothiazole]trimethincyanine, and [1-*m*-nitrophenyl-2-quinoline][1-ethyl-4-quinoline]monomethincyanine perchlorates.

A.E.S.

Cyanine Dyes containing Fluorine. IV— Cyanine Dyes from 5- and 6-Trifluoromethylthiobenzothiazoles

L. M. Yagupol'skii and M. S. Marenets

J. Gen. Chem. U.S.S.R., **25**, (9), 1771–1775 (Sept. 1955)

Work already reported (see J.S.D.C., **69**, 304 (1953)) is continued— the 5-substituted intermediate (see title) is synthesised, and six 5-substituted thiacyanines and four further 6-substituted thiacyanines are prepared. Introduction of the 5-CF_3 group results in a slight bathochromic shift (7–10 mμ.).

A.E.S.

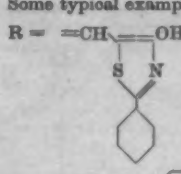
Thiazolin-4-ones (Thiazol-4-ols). II— Reactions of the Methylene Group of 2-Phenylthiazolin-4-one

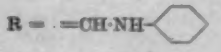
F. N. Stepanov and Z. Z. Moiseeva

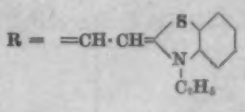
J. Gen. Chem. U.S.S.R., **25**, (10), 1977–1982 (Sept. 1955)

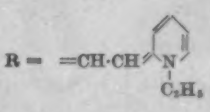
2-Phenylthiazolin-4-one (synthesised in a previous investigation— *J. Gen. Chem. U.S.S.R.*, **25**, 1170 (1955)) is converted into methin, azamethin, *mero*cyanine, and azo dyes by reaction at the reactive methylene group, the products being—

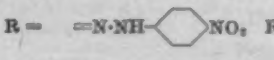
Some typical examples are—

R =  R′ = NO_2 (dark blue)

R =  R′ = NO_2 (orange-red)

R =  R′ = H (λ_{max} = 568 mμ.; a feeble photographic sensitiser to yellow-green)

R =  R′ = NO_2 (λ_{max} = 576 mμ.; a photographic desensitiser)

R =  R′ = H (orange–red in acid medium; blue in alkaline medium)

A.E.S.

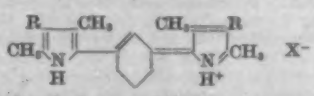
Pyrrole Dyes. I— *cyclo*Trimethin Dyes of the Pyrrole and Indole Series

A. Treibs and E. Herrmann

Annalen, **589**, 207–221 (1954)

Chem. Abs., **49**, 15854 (25 Nov. 1955)

Synthesis of various dyes of formula—



is described and of some of their analogues, e.g. the blue 3:3′:5:5′-tetramethyldipyrryl(2:2′)-*mero*-2-methyl-hexa-*cyclo*trimethin bromide.

C.O.C.

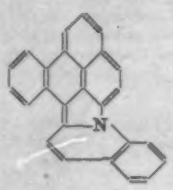
Decomposition of Quinaldic and *meso*Benzanthrone-3-carboxylic Acids in the Presence of *meso*Benzanthrone

W. Bradley and H. E. Nursten

J.C.S., 4027–4028 (Nov. 1955)

Whereas the thermal decomp. of quinaldic acid affords the 2-quinolyl anion and this reacts with quinoline to form 2:2′-diquinolyl (Brown and Hammick, *J.C.S.*, 173 (1949)),

the same decomp. in the presence of *meso*benzanthrone, which is known to undergo substitution *ortho* and *para* to the CO group, did not take the expected course, but appeared to give a pyrrocoline deriv. (I); the expected products, 4- or 6-2′-quinolyl*meso*benzanthrone, were not encountered.



(I)

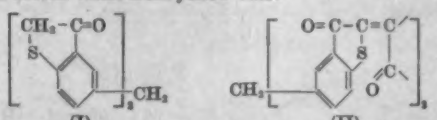
H.H.H.

Indigoid Dyes. XVI—Thioindigoid Dyes from Diphenylmethane-4:4′-disulphonic Acid

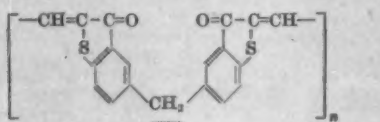
P. C. Dutta and D. Mandal

J. Indian Chem. Soc., **32**, 497–500 (Aug. 1955)

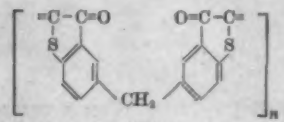
Diphenylmethane-4:4′-dithioglycollic acid, prepared from diphenylmethane-4:4′-disulphonic acid, on ring closure gave the corresponding bisthioindoxyl compound (I). This has been condensed with *o*-diketones such as isatin, its nitro and bromo derivatives, acenaphthaquinone and its nitro derivatives, phenanthraquinone, and aceanthraquinone to give thioindigoid dyes of general formula II and with glyoxal to give the product III. Oxidation of the indoxyl compound with potassium ferricyanide gave the bisindigo compound IV. These thioindigoid compounds dye cotton violet from yellow vats.



(I) (II)



(III)



(IV)

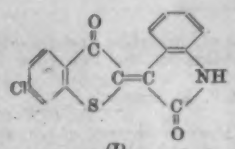
C.H.R.

Indigoid Vat Dyes of the Isatin Series. VIII—3-Indole-2′-(-6′-chloro)-thionaphthen-indigos

S. K. Guha and J. N. Chatterjea

J. Indian Chem. Soc., **32**, 551–556 (Aug. 1955)

5-Chloro-2-carboxyphenylthioglycollic acid, obtained from 4-chloroanthranilic acid, on ring closure gave 6-chloro-3-hydroxythionaphthen. This has been condensed with isatin and its chloro and nitro derivatives to give the corresponding 6′-chlorothioindigoid dyes (I), which dye cotton red from yellow vats and wool from a suspension in dilute sulphuric acid. The colours are bathochromic in comparison with those from the corresponding 5′- and 7′-chloro compounds. Oxidation of the thionaphthen compound gave 6:6′-dichlorothioindigo (Ciba Red B).



(I)

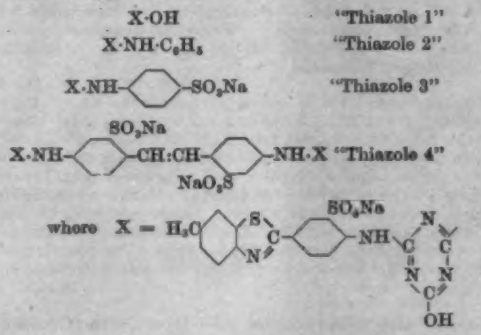
C.H.R.

Synthesis of Fluorescent Brightening Agents

S. Bartkowicz

Zesz. Nauk. Politech. Łódz., No. 2 (*Chemia* 1), 41–68 (1954)

The synthesis of fluorescent brightening agents of the following composition is described—



All four compounds have very good affinity for cotton, show better intensity of fluorescence and washing fastness than some commercial products, and have similar fastness to light.

T.Z.W.

Chlorophyll Spectra and Molecular Structure

J. C. Goedheer

Nature, **176**, 928–929 (12 Nov. 1955)

The directions in which linearly polarised light is preferentially absorbed in a pigment molecule correspond to the directions of max. transition probability of the electronic transitions which give rise to the different absorption bands in the spectra. The investigation of these directions by the study of dichroism and polarisation of fluorescence for chlorophyll a and b, bacteriochlorophyll, and the corresponding phaetophytins, is reported. The spectra are reproduced and discussed.

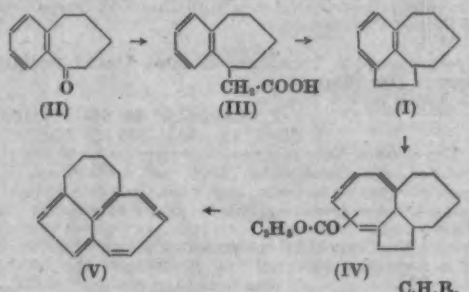
J.W.D.

Azulene and Related Substances. VIII—1:8-Tetramethyleneazulene

S. Dev

J. Indian Chem. Soc., **32**, 513–524 (Aug. 1955)

1:7-Tetramethyleneindane (I), obtained from benzosuberone (II) via benzo*cyclo*heptylacetic acid (III), gave with ethyl diazoacetate the *cyclo*heptatrienecarboxylic acid derivative (IV), which on dehydrogenation–decarboxylation gave 1:8-tetramethyleneazulene (V). The light absorption characteristics of this compound are in agreement with those calculated for 1:8-dialkylazulenes.



(II) (III) (I) (V) (IV)

C.H.R.

Resonance Force Theory of Carotenoid Pigments

W. T. Simpson

J. Amer. Chem. Soc., **77**, 6164–6168 (5 Dec. 1955)

A theory of the electronic spectra of carotenoid pigments is given in which the double bonds are treated as quasi-independent weakly interacting systems. The excited states are described as derived from an *n*-fold degenerate manifold (*n* = number of double bonds). These states split under a perturbation patterned after simple electrostatic resonance interaction. Consideration is given to the experimentally observed positions of energy levels, ground-state steric configuration, isomeric atom processes, intensity and intensity changes.

C.O.C.

Browning of Foods

V— Isolation and Identification of the Compounds involved in the Discoloration of Potatoes

Y. Obata and S. Sakamura

J. Agr. Chem. Soc. Japan, **27**, 766–769 (1953)

L-Tyrosine, chlorogenic acid, and a related compound are the prepigments involved in the darkening of white potatoes by treating with tyrosinase *in vitro*.

VI— Photo-oxidation Products of L-Tyrosine and the Mechanism of its Colour Development

Ibid., 769–772

Exposure of solutions of L-tyrosine (I) to sunlight resulted in formation of melanic pigments. 3:4-Dihydroxyphenylalanine (II) was detected by spraying a paper chromatogram of photo-oxidised (I) soln. with Folin's phenol reagent. Formation of II was highest at pH 4–5 but colour development was greatest at pH 6. II was stable to sunlight at pH 5, but markedly decomposed at pH > 6 with formation of melanin. Thus melanisation of I seems to proceed as I → II → melanin. The whole process was completely inhibited by adding 0·1% thiourea but the melanisation of II was not affected; thus thiourea seems to inhibit only formation of II from I. C.O.C.

Paramagnetic Resonance of Ultramarine

D. M. Gardner and G. K. Fraenkel

J. Amer. Chem. Soc., **77**, 6399–7000 (5 Dec. 1955)

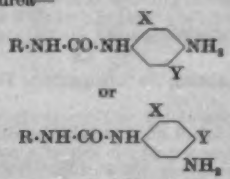
That ultramarine is blue has been generally attributed to the presence of sulphur but the nature of the sulphur component has never been elucidated. An analogy between the colour of ultramarine and the colour of solutions of sulphur in oleum has been suggested. Such oleum solutions are paramagnetic and it is shown that both natural and synthetic ultramarine show paramagnetic resonance absorption, the observed intensity being the correct order of magnitude to be accounted for by sulphur radicals. C.O.C.

PATENTS

Bluish-red to Blue Azoic Dyes for Acetate Rayon and Nylon

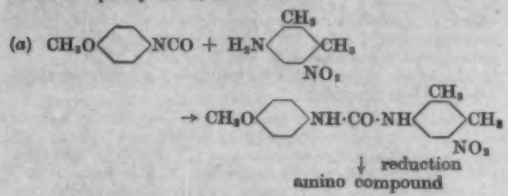
FH *BP* 742,260

Azoic dyeings of good fastness to cross-dyeing are produced on acetate rayon and nylon by applying an amino-diphenylurea—

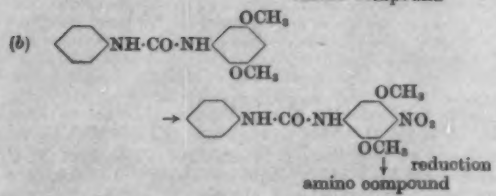


or

(R = Aryl of benzene series; X and Y = Alk, O. Alk, or Hal, or one of them may = H) together with an arylide of 3:2-hydroxynaphthoic acid or of its 7-bromo- or 7-methoxy-derivatives, diazotisation and coupling being brought about on the fibre by treatment with nitrous acid. The amino-diphenylurea compounds may be made by reacting phenyl isocyanates with suitable aminonitro compounds, followed by reduction, or by nitration and reduction of suitable diphenylureas, as follows—



(*a*) → reduction → amino compound



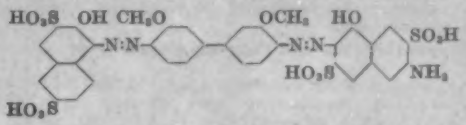
(*b*) → reduction → amino compound

Thus the 4-amino-2:5-dimethoxydiphenylurea formed in reaction (*b*) is applied to acetate rayon along with the sodium salt of the 4-methoxy-2-methylanilide of 3:2-hydroxynaphthoic acid from a slightly alkaline bath containing ammonia. The material is then treated in a cold nitrous acid bath, then at 60°C. in aq. sodium acetate and finally soaped at 85°C. to give a navy blue. E.S.

Blue Metal(Copper)-complex Disazo Direct Dyes

ICI *BP* 742,731

The copper complexes of disazo dyes made by coupling tetrazotised 3:3′-dimethoxy(or diethoxy)benzidine under alkaline conditions with 1 mol. of R acid and 1 mol. of 2-amino-5-naphthol-1:7- or -3:7-disulphonic acid or an *N*-benzoyl derivative thereof, which may be substituted in the phenyl radical by CH_3, CF_3, Hal, or O. Alk, are blue direct dyes whose light fastness is maintained when the dyed materials are subjected to a crease-proofing treatment e.g. with a urea-formaldehyde resin. The *N*-benzoyl- and subst. benzoyl derivatives required are prepared by reacting the parent aminonaphtholdisulphonic acids with the appropriate benzoyl halide in presence of an organic base (e.g. pyridine), followed by a mild alkaline hydrolysis of any *O*-benzoyl groups which may have formed. Thus tetrazotised dianisidine is coupled first with 1 mol. of R acid, and then with 1 mol. of 2-amino-5-naphthol-3:7-disulphonic acid in aqueous ammoniacal pyridine to give—

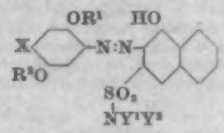


Heating with ammoniacal copper sulphate leads to dealkylation of the OCH_3 groups and formation of the copper complex, which dyes viscose rayon reddish-blue. E.S.

Blue Metal(Chromium)-complex Monoazo Dyes for Wool and Nylon

Gy *BP* 741,638

The metal complexes containing 1 atom Cr to 2 mol. of monoazo compounds

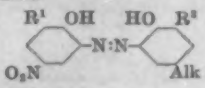


(R^1 and R^2 = Alk or preferably hydroxyalkyl of a total of < 5 C; Y^1 = H, Alk, cycloalkyl or phenyl; Y^2 = H or Alk, or Y^1 and Y^2 may be bound in the form of a ring by themselves or with an O atom) dye wool and nylon blue from neutral or weakly acid baths. Thus the monoazo compound 2:5-dimethoxyaniline→1-naphthol-3-sulphondimethylamide is heated in acetamide at 140–145°C. with chromic acetate for ca. 90 min. The chromium complex is then converted by treatment with NaOH into its soluble Na salt, which dyes wool full reddish-blue from a neutral or weakly acid bath. E.S.

Brown Metal(Chromium)-complex Monoazo Dyes for Wool, Nylon, etc.

S *BP* 741,635

The metal complexes containing 1 atom Cr to 2 mol. of monoazo compounds



(R^1 = H, CH_3 or Cl, preferably H; R^2 = H, CH_3, NH·COH, NH·COOAlk, NH·COOAlkoxyalkyl, or preferably NH·CO·Alk) dye wool, nylon, silk and leather brown from neutral baths. Thus the monoazo compound 2-amino-4-nitrophenol→2-acetylamino-4-methylphenol is boiled for 24 hr. with chrome alum, and converted into the soluble Na salt by treatment with NaOH and NaCl. E.S.

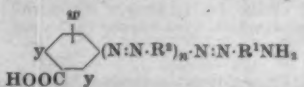
Copperable Monoazo Direct Dyes having an Anthraquinone Residue and a Triazine Residue
S *BP* 740,660

Monoazo compounds of general formula—

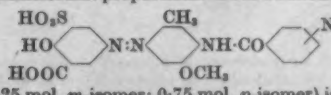
(R^1 and R^2 = residue of benzene, stilbene, or benzanilide compound, R^2 being preferably a stilbene residue; X = direct link, or a radical $-CO\cdot C_6H_4\cdot NH-$; Z = Hal, OH, NH_2 or the residue of an amine; the ring A may contain sulpho groups and/or Hal atoms; the ring B may contain e.g. an SO_3H group) are direct dyes which may be coppered in substance or on the fibre. Thus cyanuric chloride is condensed first at 0–3°c. with 1 mol. of the monoazo compound 5-sulphoanthranilic acid→1-[4″-aminostilbenyl-(4′)]-3-methyl-5-pyrazolone-2′:2″-disulphonic acid, and then at 40–45°c. with 1 mol. of 1-amino-4-*p*-aminoanilino-anthraquinone-2-sulphonic acid. The third Cl atom of the cyanuric ring is then replaced by condensation with aniline at 90–95°c. The product dyes cotton green, which may be aftercoppered. Or the copper complex may be prepared in substance by heating at 90–95°c. with ammoniacal copper sulphate. E.S.

Copperable Dis- and Poly-azo Direct Dyes
S *BP* 740,715

Extension of *BP* 698,008 (J.S.D.C., **70**, 30 (1954)). An unsaturated dicarboxylic acid dihalide X·CO·C(A):CH·CO·X (X = Br or Cl; A = H, CH_3, Br, or Cl) is condensed with 2 mol. of an aminoazo compound



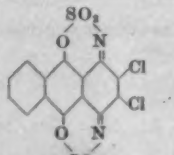
(R^1, R^2 = radicals of benzene, diphenyl, stilbene, naphthalene, pyrazolone, or acetoacetylaminoaryl series; one y = OH, the other y = H; *w* = H, Hal, Alk, NO_2, acylamino, or SO_3H; *n* = 0 or 1), to give direct dyes which may be coppered in the dyebath or on the fibre. Or mixtures with desirable properties may be produced by using a mixture of such an aminoazo compound with any other suitable aminoazo compound, not necessarily in equimolecular proportions. Thus a mixture of the 2 isomers



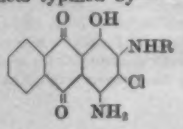
(1·25 mol. *m*-isomer; 0·75 mol. *p*-isomer) is dissolved in dil. aq. NaOH, cooled with ice, and mixed with *NN*-dimethyl-aniline. A soln. of maleic acid dichloride in *o*-dichloro-benzene is slowly added. The product dyes cotton yellow by the aftercoppering process. E.S.

β-Substituted Anthraquinone Derivatives
FBy *BP* 738,982

The product of treating 1:4-diamino-2:3-dichloro-(or dibromo) anthraquinone with oleum viz.—



is further treated with (i) aq. alkali (ii) ammonia (iii) an alkyl- or aryl-amine or (iv) a thioalcohol or thiophenol. One of the halogen atoms is thereby replaced to give, after saponifying, products typified by—



Thus the product of 1:4-diamino-2:3-dichloroanthraquinone and oleum is heated on the water-bath with 20% ammonia and after separation the resulting compound is saponified with H_2SO_4 to yield 2-chloro-1:3-diamino-4-hydroxyanthraquinone. R.K.F.

Aminoanthraquinone Disperse Dyes
Ciba *BP* 743,220

An aliphatic amine, with or without NH_3, is condensed with 1:4-dihydroxyanthraquinone in the presence of up to an equal weight of a leuco-1:4-disubstituted anthraquinone, followed by oxidising, to give blue disperse dyes. Thus, a mixture of 1:4-dihydroxyanthraquinone (80 parts) and leuco-1:4-dihydroxyanthraquinone (25 parts) is heated under pressure for 17 hr. at 60–100°c. with β-hydroxyethyl-amine and NH_3 in methanol. Sodium 3-nitrobenzene-1-sulphonate dissolved in water is added and heating continued at 100°c. for 4 hr. R.K.F.

1-Amino-6(or 7)-alkylsulphonylanthraquinones
Ciba *BP* 738,650

A β-alkylsulphonylanthraquinone (Alk of $\geqslant$5 C) is mononitrated, and the resulting mixture of 1-amino-6-alkylsulphonyl- and 1-amino-7-alkylsulphonyl-anthraquinones reduced with a water sol. hydrosulphide in such a way that the 1-amino-6-alkylsulphonyl derivative only is obtained. The 7-alkylsulphonyl compound is reduced to a 1-amino-7-mercaptoanthraquinone which may be recovered, alkylated and oxidised to the corresponding 7-alkylsulphonyl derivative. Thus, 2-methylsulphonyl-anthraquinone is nitrated in H_2SO_4 at 100°c. It is separated by adding water and filtering. The product is reduced with aq. NaHS at 64–67°c. for 5 hr. 1-Amino-6-methylsulphonylanthraquinone is filtered off. The 7-mercapto compound is separated by adding NaCl to the filtrate, methylated with CH_3I and oxidised e.g. in acetic acid with H_2O_2. R.K.F.

Indanthrone from 1-Aminoanthraquinone
FBy *BP* 741,341

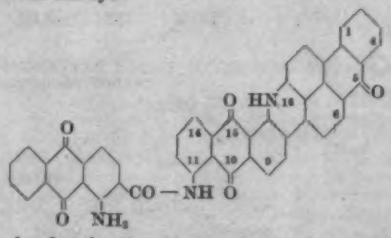
In the preparation of indanthrone described in *BP* 703,377, in which 1-aminoanthraquinone is heated in a melt of alkali phenate, the oxidising agents then used are replaced by air and a catalyst e.g. MnO_2 or iron chloride. Thus, 1-aminoanthraquinone is heated at 195–220°c. with intensive stirring for a total of 6 hr. in a melt of KOH and phenol, containing Na acetate, $MnSO_4$ and $CuSO_4$. R.K.F.

Sulphur-containing Anthraquinone Derivatives
BASF *BP* 739,250

A halogen-containing anthraquinonoid compound is heated with an alkali sulphide with or without S, in a melt of at least one water soluble salt of a carboxylic acid, to produce S containing derivatives. Thus 3-chlorobenzanthrone is heated at 140–145°c. for 4–5 hr. with Na_2S and S in a melt of Na and K acetates. The resulting 3:3′-dibenzanthronyl sulphide separates on diluting the melt with water. R.K.F.

Acylamino-Benzanthrone-Anthraquinone-Acridines — Vat Dyes
ICI *BP* 741,312

11-Amino- or 3:11-diamino-benzanthrone-3′(N):4′-anthraquinone-1″(N):2″-acridone is condensed with the halide of 1-aminoanthraquinone-2-carboxylic acid (or the corresponding 1-nitro or 1-chloro compound followed by subsequent replacement of the NO_2 or Cl by NH_2). The products are brown vat dyes suitable for applying at 85–100°c. Thus the dye—

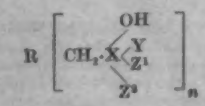


is made by first heating 11-aminobenzanthrone-3′(N)-4′-anthraquinone-1″(N):2″-acridone with 1-nitroanthraquinone-2-carboxylic acid chloride in *o*-dichlorobenzene

at 175–180°C. After filtering, the product is suspended in fresh *o*-dichlorobenzene at 180°C. and NH_3 passed through. R.K.F.

Dibenzanthrone Vat Dyes
General Aniline *USP* 2,703,321

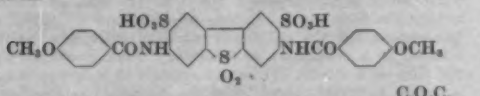
Chloromethylated vat dyes (e.g. produced by treating vat dyes with bis-chloromethyl ether in presence of conc. H_2SO_4) when treated with a hydroxyaryl compound yield dyes of formula—



(R = a vat dye nucleus; n = 1–4; X = an aryl nucleus; Y = H or OH; Z^1 and Z^2 = H, Alk, alkoxy; OH is *o*- or *p*- to the CH_2 bridge). Their properties vary widely depending on the nucleus and the value of n. Some are useful as vat dyes, e.g. dibenzanthrone and *iso*dibenzanthrone chloromethylated and condensed with phenol. Despite their free OH groups these dyes have excellent fastness to light and very good fastness to washing. Dyes of relatively low mol. wt. and in which n = 2–4 are soluble in spirit and ketone solvents but insoluble in hydrocarbons. C.O.C.

Sulphonated Dibenzothiophen Dioxide Derivatives—Fluorescent Brightening Agents
American Cyanamid Co. *USP* 2,702,759

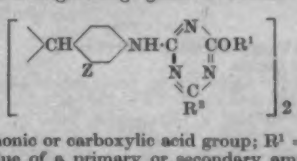
The sulphonic acids of acylated 3:7-diaminodibenzothiophene dioxide and its nuclear substitution products are fluorescent brightening agents having good affinity for fibres, and good physical and chemical stability. Thus dry 3:7-diaminodibenzothiophene-5:5′-dioxide-2:8-disodium sulphonate (3 parts by wt.) is stirred with *p*-anisoyl chloride (7·7) and pyridine (0·25–0·5), heated to 150–160°C., cooled, diluted with acetone, filtered, washed, suspended in dil. hot aq. Na_2CO_3, filtered, washed and dried. The light brown product yields a strongly blue fluorescent solution in water and is substantive to cellulose. It has the formula—



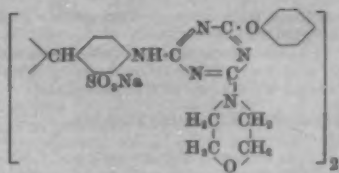
C.O.C.

Fluorescent Brightening Agents
General Aniline *USP* 2,703,801

Fluorescent brightening agents of formula—



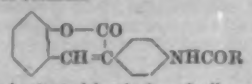
(Z = sulphonic or carboxylic acid group; R^1 = Alk or Ar; R^2 = residue of a primary or secondary amine or of an alcohol or phenol) are obtained by treating a 4:4′-diaminostilbene sulphonic (or carboxylic) acid with cyanuric chloride and treating the resulting triazyl derivative with 2 mol. of a phenol or alcohol and then with 2 mol. of an alcohol, phenol or a primary or secondary amino compound. Thus cyanuric chloride (44·4 parts) in acetone (230) is poured quickly into ice and water (600). Diaminostilbenedisulphonic acid (44·4) in water (400) is added together with sufficient Na_2CO_3 to neutralise the sulphonic groups. The mixture is kept neutral to Congo Red and at < 5°C. until no more diaminostilbenedisulphonic acid is present as determined by a tetrazotisation and coupling test. To the resulting solution or suspension of [2:4-dichloro-1:3:5-triazyl-(6)-]diaminostilbenedisulphonic acid sodium salt is added phenol (22·5) in water (100) and then 40% NaOH (25). After several hours stirring at 5–10°C. and several more hr. at room temperature, morpholine (29) is then added followed by several hr. stirring at 60–70°C., the mass being kept alkaline by addition of Na_2CO_3. The product is cooled to room temperature, filtered and dried. It has the formula—



and has very strong bluish fluorescence, it is substantive to cotton and applied either directly or mixed with a detergent, it does not have high build-up properties so that repeated washings in detergents to which it has been added do not result in undue brightness nor does the fabric become discoloured due to deterioration of the brightening agent. C.O.C.

Fluorescent Brightening Agent
DuP *USP* 2,702,296

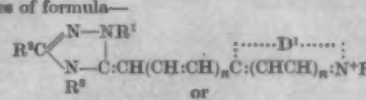
Compounds of formula—



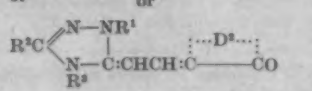
(R = phenyl substituted by 1, 2 or 3 alkoxy radicals, one methyl and one methoxy radical, or a methylenedioxy radical), e.g. 3-*p*-(3-methoxybenzamido)-phenylcoumarin, have good affinity for cellulosic and hydrophobic fibres on which they show blue fluorescence, fast to light and bleaching. C.O.C.

Cyanine Dyes
Ilford *BP* 743,133

Dyes of formula—



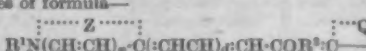
or



(R^1 = Alk or subst. or unsubst. Ar; R^2 = Alk, alkylthio or aralkylthio; R^3 = Alk or hydroxyalkyl; R^4 = Alk; X = acid radical; n = 0 or 1; D^1 = atoms to complete a 5- or 6-membered ring; D^2 = atoms to complete a nitrogen ketomethylene nucleus), e.g. (1:3:4-trimethyl-1:2:4-triazole - 5 -)(3′ - methylbenzthiazole - 2′)monomethincyanine perchlorate, are photographic sensitisers. They are prepared by reacting together appropriate triazole quaternary salts and heterocyclic nitrogen compounds. C.O.C.

***mero*Cyanine Dyes containing a Chain Substituent**
Kodak *BP* 742,206

Dyes of formula—



(R^1 = Alk or hydroxyalkyl; R^2 = Alk of 1–4 C; Z = atoms to complete a 5- or 6-membered ring; n and d = 0 or 1; Q = atoms to complete a nucleus of the 5-thiazolone or 5-oxazolone series), e.g. 4-[α-ethoxy-β-(1-ethyl-2-quinolylidene)ethylidene]-2-ethylthio-5-thiazolone, can be used as photographic sensitisers. C.O.C.

Carbon Black
Godfrey L. Cabot *BP* 743,488

Modification of *BP* 699,406 (J.S.D.C., 70, 30 (1954)) by using as the hydrocarbon anthracene oil of average mol. wt. 140–225.

BP 743,523

A detonation method for obtaining an extremely fine and black pigment.

BP 743,879

A method for producing carbon black of exceptionally fine particle size. C.O.C.

Nacreous Lead Phosphate Pigment
Sherwin-Williams Co. *USP* 2,703,747

A lamellar crystalline pigment, which used in aqueous suspension resembles pearl essence, is produced by mixing at 70–90°C. an aqueous alkali metal monoacid phosphate

(½ mol. per litre) with aqueous lead nitrate (0·5 mol. per litre). The lead nitrate solution is brought to pH 1–3, preferably 1·5, with nitric acid before being mixed with the phosphate solution. There should be a stoichiometric excess of about 4% by wt. of the phosphate. C.O.C.

Water-dispersible Titanium Dioxide

National Lead Co. *BP* 742,500

Rutile TiO_2 is calcined, wet ground, and 0·1% (on wt. of pigment) of formic or acetic acid added to flocculate the pigment. The flocculent pigment is then heated at 110–180°C. to dry it and evaporate off all the volatile acid. Finally it is milled to yield a water-dispersible pigment. C.O.C.

Titanium Dioxide Pigments for use in Lithographic Inks

American Cyanamid Co. *BP* 743,629

Coating the particles of TiO_2 with a hydrophobic organophilic thermocured resinous product obtained by treating an alkylated methylol melamine with an *N*-alkylol fatty acid amide of chain length 12–20 C renders the pigment particularly useful for making lithographic inks or the like. The pigment is coated with the resinous precondensate and then heat cured.

BP 743,630

If TiO_2 particles are first coated with a water-insoluble polyvalent metal hydrous oxide, e.g. $Al(OH)_3$ and then with a polyorganosiloxane followed by heat curing, they are given great resistance to wetting by water and to attack by acids. The coated pigment is especially suitable for lithographic and other inks for use in planographic printing. C.O.C.

Buff-red Zinc Oxide

Merck & Co. *BP* 742,452

Buff-red zinc oxide of particle size < 300 A. and very hygroscopic is obtained by heating ammoniated zinc carbonate at 100–400°C. The product is well suited for making zinc ferrite or for other purposes where zinc oxide of extremely fine particle size is desired. C.O.C.

Efficiency of Chromate Pigments as Anti-corrosion Agents (V below)
4′-Nitroazobenzene-4-carboxyl Chloride as Reagent for Alcohols (XIV p. 132)
Chromatography of Dye Intermediates (XIV p. 132)

V— PAINTS; ENAMELS; INKS

Technical Developments in Pigments and Paints during 1955

Anon.

Paint, Oil & Colour J., 129, 73–77 (6 Jan. 1956)

Brief reviews are given under the following headings—Pigments and Paints, Varnishes, Oils, Resins, Plant and Equipment, Analysis and Testing, Printing Inks, Corrosion and Weathering. 121 references. C.O.C.

Efficiency of Chromate Pigments as Anti-corrosion Agents

G. W. Grupp

Org. Finishing, 16, (7), 14, 15 (1955)
Chem. Abs., 49, 16455 (25 Nov. 1955)

On three different types of Mg alloy, one mild steel and one Al alloy, out of 20 different paints applied followed by exposure including thrice daily spraying with sea water, best all round protection was given by $CaCrO_4$, $SrCrO_4$ and Zn monoxy chromate. Good protection was given by K Zn chrome and Zn tetroxychromate. Fair protection was given by Na chrome. All the others gave poor protection against corrosion. C.O.C.

Protective Action of Pigments on Steel

J. Electrochem. Soc., 101, 141–148 (1954)
Chem. Abs., 49, 16456 (25 Nov. 1955)

Aqueous extracts of litharge, metallic lead, red lead, white lead, zinc and zinc oxide, both under aerated and deaerated conditions, were tested as inhibitors for the corrosion of steel. Litharge extracts inhibited corrosion completely, presumably because of the presence of $Pb(OH)^+$ ions. Extracts from lead and red lead inhibited for a short time, whereas extracts from white lead, zinc and zinc oxide had no effect. Protective properties of the extracts were related to their reserve alkalinities. Extracts of litharge, lead and red lead gave protection only under aerated conditions, under deaerated conditions they slowly attacked steel. The passive film on steel formed with the litharge extracts was γ-Fe_2O_3. The lead extracts contained massive and colloidal $Pb(OH)_2$. C.O.C.

Effect of Various Extenders, especially Wollastonite, in Alkyd Flat Finishes

F. King

Offic. Dig. Federation Paint Varnish Production Clubs, 27, 15–23 (1955)
Chem. Abs., 49, 16455 (25 Nov. 1955)

Wollastonite, an almost pure Ca metasilicate, is an extender pigment giving desirable properties to various paints. It is superior to other types of inerts in alkyd flat wall paints having equal flatting power to the others and lower binder demands. In the enamel hold out test to determine film porosity, paints containing wollastonite have a high maximum useful polyvinyl chloride value. C.O.C.

Passage of Water through a Paint Film

H. G. Rains and G. L. E. Wild

J. Oil & Col. Chem. Assocn., 38, 800–802 (Dec. 1955)

Doubt is expressed as to the existence of "pores" or capillaries such as are commonly thought to assist the penetration of a paint film by water. Relative adhesion of film and of water to pigment may play a major rôle, with the degree of internal rigidity of the film acting as a complicating factor. It is proposed that the initial penetration of the matrix by water is a "solvent"–"solute" process akin to that of the initial swelling of rubber by benzene. Water would thus enter the film under the stimulus of secondary valency forces, within limitations imposed by mol. size and shape; the appropriate summation of these factors gives, in principle, the free-energy change of the water–film interaction. The energy of association of OH groups in hydroxylated films such as alkyds would be quite high. Curves are reproduced, showing the change in water absorption of a linseed alkyd paint film, and of the corrosion of the steel substrate, with variation in pigment (iron oxide and Paris white) volume fraction (PVF). Both curves show clear minima at a PVF of 0·6–0·7. As this PVF level is approached the vol. fraction of medium within the film which remains unorientated by multilayer adsorption at the pigment surface decreases rapidly, leading to a reduction in secondary valency reactivity in the film as a whole. This corresponds to a decrease in reactivity towards water, and an increase in mechanical rigidity against penetration. When the PVF exceeds 0·6–0·7, both water absorption of the film and corrosion of the steel increase rapidly; thus, as the pigment voids become less well filled, the porosity of the film assumes control. J.W.D.

PATENTS

Printing Ink and Varnish therefor

J. M. Huber Corpn. *BP* 742,665

A varnish for rapid drying, press stable inks consists of a liquid vehicle and a salt of a water-insoluble resin of acid number $\not< 90$ and a nitrogenous base of dissociation constant $> 1 \times 10^{-9}$. The vehicle contains either an alcohol, formamide, a glycol, a glycol ester or a glycol ether. C.O.C.

Stencil Duplicating Ink

A. B. Dick Co. *BP* 742,301

The ink consists of a water-soluble dye, and/or water-dispersible pigment, a wetting agent, an organic resinous material containing hydroxy and/or amino groups to body the material and act as an adherent base upon drying, and a liquid polyhydric alcohol, the whole being diluted with water.

BP 742,357

The organic resinous material is polyvinyl alcohol, a urea-aldehyde or phenol aldehyde precondensate, a polyethylene glycol of > 29 C, a carbohydrate or protein. There is also present an aldehyde which is inactive while in dilute solution. C.O.C.

Classification of Pigment Particles for Paints

British Ceca Co. *BP* 736,590

A pulverised pigment is suspended in an inert gas and passed through a cyclone separator to remove oversize particles, the refined suspension is fed coaxially with an atomised solvent or paint vehicle to wet and aggregate the

pigment and the wetted particles are removed from the gas stream in a second cyclone separator for incorporation with the remaining components of the paint, lacquer, enamel etc. E.C.

Water-base Paints
General Electric Co. *BP* 743,685

Incorporation into a water-base paint, e.g. a casein or rubber latex paint, of a little of a water-soluble alkali-metal salt of a hydrocarbon-substituted silane triol results in the dried paint films having improved washability and improved resistance to staining by aqueous media. C.O.C.

Barium Compounds for preventing Water-dispersion Paint Films from Blooming
Firestone Tire & Rubber Co. *USP* 2,702,284

Water paints containing non-rubbery or resinous synthetic polymers often yield films which sooner or later show bloom. This can be avoided by adding a barium compound to the paint so that there is about 0·5 g. Ba ion present per 100 g. of polymer. Suitable compounds are $BaCO_3$, Ba malonate, Ba stearate or Ba citrate. C.O.C.

Dispersions of Polytrifluorochloroethylene
M. W. Kellogg Co. *BP* 737,267

Dispersions of polytrifluorochloroethylene are prepared by ball-milling the ground polymer in a mixture of an ester, ketone or ether-alcohol with an aromatic hydrocarbon, e.g. di-isobutyl ketone and xylene, until the particle size is approx. 1 μ. The dispersions may be applied to surfaces by dipping or spraying and give continuous films after fusion, e.g. at 370–400°C. E.C.

Corrosion-resistant Primers
Pinchin, Johnson & Associates *BP* 735,883

Adherent corrosion-resistant primers for ferrous metals comprise a suspension of zinc tetroxychromate in a solution of a partially hydrolysed (hydroxyl value 2–10%) copolymer of vinyl chloride (50–80%) and vinyl acetate (50–20%) and phosphoric acid in a suitable solvent mixture. E.C.

Water Adsorption on Polystyrene, Cellulose Triacetate, and Polyvinyl Chloride below Saturation Vapour Pressure (XIII p. 128)
Toluidine Red Bloom in Stoved Synthetic Finishes (XIII p. 129)

VI—FIBRES; YARNS; FABRICS

Static Electrification
D. F. Arthur

J. Textile Inst., **46**, T 721–T 734 (Dec. 1955)

A comprehensive survey of static electrification, including 67 references. J.W.B.

Friction between Dissimilar Fibres
J. Mazur

J. Textile Inst., **46**, T 712–T 714 (Nov. 1955)

Static friction between single fibres is measured by the inclined-fibre and slip-stick methods, using dissimilar fibres. Results are compared with earlier data obtained with similar pairs of fibres (H. G. Howell and J. Mazur, *ibid.*, **44**, T 59 (1953)) and are expressed graphically according to the relation $F = \alpha R^n$ where R = normal load, F = frictional force, and α and n are constants. The plots are in all cases linear, though there appears to be no general relation between the values of the constants for dissimilar pairs and those for the corresponding similar pairs. J.W.B.

Effect of Swelling and Supermolecular Structure on Reaction of Cellulose with Nitrogen Dioxide
W. E. Roseveare and D. W. Spaulding

Ind. Eng. Chem., **47**, 2172–2175 (Oct. 1955)

Dilute solutions of nitrogen dioxide in carbon tetrachloride react preferentially with the more accessible amorphous regions of cotton and viscose rayon. At high concentrations, however, the nitrogen dioxide penetrates the crystallites, and then the crystalline and amorphous cellulose react at the same rate. A much higher concentration is required to penetrate the crystallites of cotton than those of rayon. Moisture regain and the stress–strain properties of the oxycelluloses produced depend on the relative proportions of amorphous and crystalline materials that have reacted. W.K.R.

Relation of the Non-cellulosic Constituents of Flax Fibre to its Spinning Quality
J. F. Couchman

J. Textile Inst., **46**, T 742–T 758 (Dec. 1955)

Several grades of flax are analysed to determine the relation, if any, between spinning quality and non-cellulosic material. Water- and NaOH-solubles, uronic anhydrides, lignin, cuprammonium fluidity, moisture regain, and acetyl and free COOH group contents are determined. There are no fundamental differences between the components of each soluble fraction, but the amounts vary and the variations are greatest in the more easily soluble fractions. Water-solubles appear to be held on the surface by hydrogen bonds, and the fraction soluble in 0·02 N-NaOH contains the highest free OH content. This fraction probably forms a network of side-chains in the spaces of the amorphous region. High-grade fibre shows generally low values for water-solubles, water-soluble uronic anhydride, lignin soluble in sodium chlorite, and fluidity. Separation of NaOH-solubles into four fractions indicates that high values for the first (0·02N-) soluble fraction and low values for the remainder are associated with good quality. In poor grades, degradation and oxidation of the hexosan (probably glucosan) in the 0·02 N-NaOH-soluble fraction to uronic anhydride has increased the water-solubles and decreased the NaOH-solubles. This has the undesirable effect of causing harshness on the surface and a more rigid alignment in the amorphous regions, thus reducing flexibility. In extreme cases main-chain degradation is shown. J.W.B.

Chain Length and Chain-length Distribution of Untreated Cotton, Flax, and Ramie Celluloses
T. E. Timell

Ind. Eng. Chem., **47**, 2166–2172 (Oct. 1955)

Values of 5750, 4700, and 4650 were obtained for the average D.P. of ramie, cotton, and flax celluloses respectively, by determination of the intrinsic viscosities of their nitrates in ethyl acetate with corrections for the influence of rate of shear and degree of substitution. The nitrates were fractionated by precipitation from acetone solution by addition of water. Prolonged refractionation was prohibited by the instability of the nitrates. In each case the frequency distribution contained only one peak, that of ramie being almost symmetrical with most of the material within 4500–6000 D.P. W.K.R.

Kinetics of the Reaction between Carbon Disulphide and Caustic Soda—II
B. Philipp

Faserforsch. und Textiltech., **6**, 509–520 (Nov. 1955)

The course of the reaction between carbon disulphide and caustic soda and the effects of additions of oxidising and reducing agents, alcohols of low molecular weight, and certain wetting agents are studied. Sulphide, trithiocarbonate, and xanthate are estimated potentiometrically, and carbonyl sulphide, perthiocarbonate, and thiosulphate polarographically. Sulphide sulphur is estimated iodometrically, and total sulphur is precipitated as barium sulphate. The results are evaluated kinetically as far as is possible in view of the heterogeneous nature of the reaction, and are discussed with reference to the technical viscose process. W.R.M.

Shortened Viscose Processes
W. Matthaes

Faserforsch. und Textiltech., **6**, 398–401 (Sept. 1955)

A discussion of the practical application of the previously described viscose process (see J.S.D.C., **71**, 63 (Jan. 1955)), particularly with respect to various methods of accelerating the ageing of the soda-cellulose (which receives no depolymerising pretreatment and is not pressed to remove excess of caustic soda soln.) by the use of hydrogen peroxide, ammonium persulphate, sodium sulphide, or caustic soda of higher concentration. A.E.S.

Can the Properties of Viscose Rayon be Improved by Increase in Crystallinity?
K. Schwertassek

Faserforsch. und Textiltech., **6**, 351–357 (Aug. 1955)

Normal and high-tenacity viscose rayons are steamed under pressure in order to increase their crystallinity, but advantageous effects are obtained only in the highly oriented fibres, which acquire increased tensile strength, reduced alkali solubility, and more uniform dyeing

properties; normal, unoriented fibres suffer a loss in wet strength in the process. It is considered that increase in the size of the unoriented crystallites of normal fibres is a move towards the state of a crystalline powder and therefore results in loss of fibre characteristics. A.E.S.

Cellulose-solvent System Ferric Iron–Tartaric Acid–Potassium Hydroxide
G. Jayme and F. Lang
Kolloid-Z., **144**, 75–81 (Nov.–Dec. 1955)

A new solvent for cellulose, believed to be a solution of the complex $(C_2H_4O_6)_3FeK_6$ in excess of KOH soln., is described. Maximum solvent power is obtained with a composition corresponding to 305–320 g. of complex per kg. and 13–17% calculated "free" KOH. Within these limits, linters give clear green solutions stable in air for a week. The solvent power is less than that of the corresponding sodium complex. With many celluloses a certain proportion, probably highly polymerised, is undissolved. Maximum swelling occurs at a higher KOH concentration than the 13–17% "free" KOH in the solution which dissolves cellulose. It is suggested that this is due to the hygroscopicity of the complex, which binds water, leading to a higher concentration in the excess of KOH solution. W.R.M.

Combination of Wool with Acids
B. Olofsson
J.S.D.C., **72**, 19–23 (Jan. 1956)

In reply to criticism by L. Peters, comparisons between different methods of calculating and predicting equilibrium constants (pK) for exchange of Cl^- and SO_4^{2-} ions on wool are considered. The Gilbert–Rideal theory accounts for the experimental value of $pK = 0{\cdot}50$–$1{\cdot}00$, whereas the Donnan theory does not. The influence of swelling volume and ionic volumes, the meaning of $pK > 0$, the relationship between thermodynamic potential and pH, and the influence of the ionic strength are also discussed, providing further justification for the use of the Gilbert–Rideal theory. Thus the earlier conclusions of the author are in the main correct. AUTHOR

Birefringence of Keratin Fibre
S. Okajima
Mem. Fac. Technol., Tokyo Metropol. Univ., (5), 295–304 (1955)
Chem. Abs., **49**, 16441 (25 Nov. 1955)

The intrinsic birefringence (Γ) of wool is not affected by heat. Stretching in absence of heat has no effect up to 60% elongation, where Γ increases; this does not correspond to the $\alpha \rightleftarrows \beta$ keratin transformation. Stretching and then heating results in slight decrease of Γ, again not corresponding to the $\alpha \rightleftarrows \beta$ transformation. Stretching and contracting decrease Γ which, on restretching, returns almost to its original value. Change in Γ during contraction and restretching compared with lack of change when virgin fibre is stretched, is caused by breakdown of the cystine bonds during heating (contraction) with resulting disorientation of the peptide chains. It seems that it is this disorientation and not the breaking of the cystine bonds which causes the changes in Γ. C.O.C.

Free Amino Groups of Soluble Feather Keratin
A. M. Woodin
Nature, **176**, 1117 (10 Dec. 1955)

After reaction of cysteic acid–keratin with fluorodinitrobenzene the total amount of α-amino acids obtained as dinitrophenyl derivatives is only 0·1 equiv. per mole of protein and the concentration of dinitrophenyllysine 0·1–0·15 mole per mole of protein. Cysteic acid–keratin, treated with exchange resins, combines with less than 0·2 equiv. of hydroxyl ion per mole of protein between pH 6 and 8·5. Between pH 8·5 and 10·5, 0·2 equiv./mole is bound. Since the molecular weight of cysteic acid–keratin is known and the gross composition consistent with the absence of a non-protein prosthetic group, the failure of the α-amino groups to react with hydroxyl ion or fluorodinitrobenzene is evidence that the protein has a cyclic structure. W.R.M.

Microscopical Examination of Skin Effects of Man-made Protein Fibres
S. Wolf
Faserforsch. und Textiltech., **6**, 357–364 (Aug. 1955)

Various man-made modified protein fibres— Tiolan (I), Ardil B (II), Thiozell (III; no longer in production), Fibrolane (IV), and Vicara (V)— are fed to the larvae of various moths, and the excreted fibre remnants are examined microscopically. Photomicrographs illustrating the results for I and II are shown. I–IV have very resistant skins, and fibres at all stages of attack can be found, the core being degraded and dissolved until an intact hollow tube of skin remains. In I (and III?) small injuries to the skin are observed, and these often develop as action proceeds and lead to the breaking of the fibre fragments into shorter pieces. V is extremely resistant throughout, and the fragments are scarcely affected by the digestion process. A.E.S.

Analysis of the Textile Qualities of Synthetic Fibres
H. Böhringer
Faserforsch. und Textiltech., **6**, 314–328 (July 1955)

Tabular and graphical comparisons are made of the textile properties of synthetic and natural fibres, particularly in relation to the serviceability of fabrics constructed from synthetic fibres, alone or in admixture with natural fibres. A.E.S.

Skin and Allied Structural Effects in Nylon
S. C. Simmens
J. Textile Inst., **46**, T 715–T 720 (Nov. 1955)

Skin effects not previously reported are described, and a mechanism is suggested for the formation of the type of skin suggested by Woods (*ibid.*, **46**, T 629 (1955)). J.W.B.

Chemistry of the Polymerisation of 6-Hexanolactam (ε-Caprolactam)
P. H. Hermans
J. Appl. Chem., **5**, 493–501 (Sept. 1955)

It is known that, when heated, 6-hexanolactam will polymerise only when a second component, e.g. water, is present. The equilibrium state attained in the lactam–water system is studied, analyses being made for unconverted lactam, cyclic oligomers, 6-aminohexanoic acid, total chain cpd., and end groups (NH_2 and COOH). The results are analysed in terms of equilibrium const. for the formation and hydrolysis of amide groupings (K_1 for lactam amide groupings and K_2 for amide groupings in linear molecules). In accordance with Flory and Schulz's assumption in their general theory of polycondensation, K_2 is independent of chain length; also, its value is in accord with that found for 7-aminoheptanoic acid (for which lactam formation is negligible), and in both cases it decreases with increase in the water content of the system (i.e. decrease in D.P.). The results indicate that amide-bond formation and hydrolysis alone cannot account for the rate of lactam conversion; there is at least one other predominating reaction— probably addition of lactam at ends of chains. A.E.S.

ε-Caprolactam Polymerisation. VII— Kinetic Analysis of Experimental Data for Formation of Poly-ε-caprolactam by Condensation Polymerisation
F. Wiloth
Kolloid-Z., **144**, 58–74 (Nov.–Dec. 1955)

A kinetic analysis of previous experimental data is made. Reasons for differences between current theory and experiment are discussed. W.R.M.

Catalytic Polymerisation of ε-Caprolactam
S. Chrzczonowicz and J. Moniewski
Zesz. Nauk. Politech. Lódz., No. 2 (*Chemia* 1), 77–96 (1954)

Influences of various organic and inorganic compounds on the polymerisation of ε-caprolactam are compared, and that of anhydrous sodium acetate is studied in detail. It was found that this salt had a positive effect, but the polymer produced in its presence was inferior to that obtained in presence of caustic soda. The former contained 23–33% of unchanged monomer. The mechanism of polymerisation is explained. T.Z.W.

Polymerisation of ε-Caprolactam in Bulk and in Solution in the presence of a Basic Catalyst
S. Chrzczonowicz
Zesz. Nauk. Politech. Lódz., No. 9 (*Chemia* 3), 93–96 (1955)

The catalytic action of mixtures of KOH and ZnO and of NaOH and CaO has been studied. Polymerisation of caprolactam in boiling xylene in presence of Na and CO_2 gave a 30% yield. The product thus obtained was found to

have a marked positive catalytic effect on the bulk polymerisation of caprolactam and of caprolactam with 2-vinylpyridine and 6-methyl-2-vinylpyridine. T.Z.W.

Equilibrium between Polymer and ε-Caprolactam in the Polyamide Depolymerisation Process

E. Turska and J. Cypryk

Zesz. Nauk. Politech. Łódz., No. 9 (*Chemia* 3), 17–30 (1955)

Polymer, which had been purified by extraction with boiling water and dried over P_2O_5, was subjected to depolymerisation in an atmosphere of N_2 at 240°C., 260°C., and 280°C. for several hours, samples being analysed hourly. Also, ε-caprolactam, purified by vacuum distillation, was polymerised under similar conditions. The relationship between monomer and water at equilibrium was found to be complex, but water decreases polymerisation. T.Z.W.

Crystallisation of Polyamides

J. Dechant

Faserforsch. und Textiltech., **6**, 422–425 (Sept. 1955)

It is known that normal unstretched Perlon L has a hexagonal lattice and is converted into the energetically more favourable monoclinic form by stretching. This form is obtained also when the melt is cooled slowly and when the rapidly cooled melt is annealed by suitable heat treatment. These facts are investigated quantitatively by X-ray diffraction experiments; 14 X-ray pictures are shown. A.E.S.

Thermal Effects occurring during the Drawing Process

M. Hrubý

Faserforsch. und Textiltech., **6**, 309–314 (July 1955)

A general mathematical treatment is given of the relation between the thermal changes occurring in a filament undergoing irreversible stretch and various properties of the filament. In cold drawing, which is applied to fibres for which the second transition point is at low temp. (e.g. nylon), the rise in temp. resulting from the evolution of heat equiv. to the work of deformation increases the plasticity of the fibre substance and therefore reduces the work required for further deformation; the temp. then no longer rises so rapidly. A characteristic feature of the process is the formation of a very short deformation zone (neck). When stretching occurs in a gas, the cooling effect of the medium is insufficient to affect the course of the process throughout the fibre, but when a liquid medium is used the surface layers of the fibre, and therefore the properties of the finished fibre, are affected. In hot drawing (high second transition point; e.g. many vinyl copolymers) the effect of heat exchange with outside sources is predominant; the deformation zone is very long. The drawing of bundles of filaments is given some consideration. A.E.S.

End-group Titration in the Determination of the Heterogeneity of Perlon L

W. Griehl and H. Sieber

Faserforsch. und Textiltech., **6**, 329–331 (July 1955)

End-group estimations on relatively homogeneous fractions of Perlon L are correlated with viscosity determinations on the fractions, so that, when the same procedure is applied to unfractionated Perlon L, it is possible to determine the weight-average and number-average mol. wt. and hence the heterogeneity, which is found to have the value 0·6 for a number-average mol. wt. of 11,000 (Flory's theory requires 0·99). A.E.S.

Aromatics in Fibres and Films

G. P. Hoff and J. L. Martin

Ind. Eng. Chem., **47**, 2122–2125 (Oct. 1955)

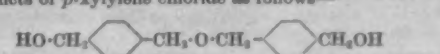
An assessment is made of the future market for fibres and films. Spectacular estimates of future growth in consumption of fibres are discounted. By the early 1960s little change is forecast in the total U.S. demand for fibres. Nylon and Dacron, the fibres based on aromatic raw materials, are expected to increase from the present 3·5% to approx. 7% of the total, whilst the proportion of all man-made fibres increases from the present 25% to 30%. Only polyester films are produced from aromatic chemicals; at present they represent only a minute part of the total film output, but their production is expected to expand considerably. W.K.R.

Terephthalic Acid from p-Xylylene Chloride

J. Manka, J. Tomaszewski, and M. Wajnryb

Zesz. Nauk. Politech. Łódz., No. 9 (*Chemia* 3), 31–43 (1955)

Oxidation of *p*-xylylene chloride with nitric acid of 71·3% concentration, at 117·5–120°C. for 3 hr., gave yields of 54·5% of terephthalic acid. Further additions of nitric acid (80%) raised the yield to 62·6% and produced the compound in 82·5% purity. Oxidation of the hydrolysis products of *p*-xylylene chloride, obtained under normal pressure, with nitric acid of 84% concn. gave 63·1% yields of terephthalic acid of 92·2% purity, which was suitable for direct esterification. The authors formulate the hydrolysis products of *p*-xylylene chloride as follows—



T.Z.W.

Synthesis of Dimethyl Terephthalate

J. Tomaszewski, M. Wajnryb, and A. Zawadzki

Zesz. Nauk. Politech. Łódz., No. 6 (*Chemia* 2), 81–89 (1955)

Esterification was carried out under atmospheric pressure, in 99% methyl alcohol, in presence of 98·7% sulphuric acid (sp. gr. 1·84), under reflux. When the relative amounts of terephthalic acid, sulphuric acid, and methyl alcohol were 1 : 4 : 15, a 99% yield was obtained after 165 min. The time of reaction increased by 35 min. when the proportions were changed to 1 : 2 : 15. Loss of methyl alcohol in recovery and reaction was 11–17%. Dimethyl terephthalate was recrystallised from ethyl alcohol, which can be recovered to the extent of 90%. The method has good prospects in industry, as was shown by pilot-plant work. T.Z.W.

Fibre Properties of Modified Aromatic Polyesters— I

W. Griehl and R. Hoffmeister

Faserforsch. und Textiltech., **6**, 504–508 (Nov. 1955)

Different possibilities of modifying aromatic polyesters, such as the formation of mixed and block polyesters and the introduction of blocks of polyether either into the main chain or as a graft, are discussed. The preparation of some mixed polyesters of polyethylene terephthalate and poly-'diol' terephthalates is described and the use of polyester mixtures and block polyesters discussed. Improved qualities, compared with those of polyethylene terephthalate, are said to be obtained, particularly with mixed polyesters involving sebacic acid. W.R.M.

Manufacture of Polyacrylonitrile for Fibre Production

F. Moll

Faserforsch. und Textiltech., **6**, 295–300 (July 1955)

Acrylonitrile is manufactured in the Buna works from acetylene and HCN, the latter being obtained by the dehydration of formamide (prepared from CO and NH_3 in presence of methanol, which can be recovered and recirculated). Some of the work carried out in establishing the conditions required for the preparation of HCN and of acrylonitrile is described. Solution and emulsion polymerisation are compared, and the relative merits of potassium persulphate and hydrogen peroxide as initiators are discussed. A.E.S.

Polyacrylonitrile Fibres

V— Effect of Molecular Weight Distribution on the Properties of Polyacrylonitrile Fibres

A. Hunyar

Faserforsch. und Textiltech., **6**, 300–309 (July 1955)

Experiments on fibres derived from polyacrylonitrile preparations varying greatly in mol. wt. distribution show that the textile properties of the fibres are not greatly dependent on the degree of homogeneity. A.E.S.

VI— Organic Solvents for Polyacrylonitrile

A. Hunyar and W. Möller

Faserforsch. und Textiltech., **6**, 442–449 (Oct. 1955)

Assessments are made of the "quality" of a number of organic solvents for polyacrylonitrile from thermodynamic considerations. Solution and gel temperatures are given for 6% solutions of the polymer, and also the variation of viscosity with concentration in relatively concentrated solutions. The results are considered in terms of the use of the solvents in spinning, and some physical data from spinning experiments are given. W.R.M.

VII—Inorganic Salt Solutions as Solvents for Polyacrylonitrile

A. Hunyar and V. Gröbe

Faserforsch. und Textiltech., **6**, 496–503 (Nov. 1955)

The viscosities of solutions of polyacrylonitrile in aqueous salt solutions have been studied in order to assess the value of the solvents. Salts used include sodium, potassium, ammonium, and calcium thiocyanates; barium, magnesium, sodium, and aluminium perchlorates; and zinc chloride. In most cases viscosity increases with concentration of salt, but in the case of sodium thiocyanate a minimum is observed at 51·5% salt. Minima are observed also when aqueous solutions of binary salt mixtures are used, and a solution of a mixture of zinc and calcium chlorides seems to be particularly effective as a solvent. W.R.M.

Chemical and Physical Behaviour of Glass Fibres in Water

R. Bennewitz, W. Bobeth, and H. Dittmann

Faserforsch. und Textiltech., **6**, 391–398 (Sept. 1955)

Leaching experiments indicate that the improvement in the wet strength of glass fibres effected by treatment with cationic resins and surface-active agents is not to be attributed to the formation of a protective film or of a film capable of taking up sodium ions liberated by the glass; in fact, previous results on the reduced loss in strength in treatments with soln. of cationic textile auxiliaries (see J.S.D.C., **69**, 469 (1953)) appear to be attributable entirely to the low pH of the soln. used. In yarns and fabrics the loss in strength is very much greater than in single fibres, and there is no completely satisfactory explanation of this fact. A.E.S.

PATENTS

Regenerated Cellulose Yarns of Improved Fatigue Resistance

DuP *BP* 741,727

Yarns of high tenacity and high fatigue resistance are produced by extruding viscose containing a soluble salt of an *N*-substituted dithiocarbonic acid into an acid coagulating bath containing 1–15% zinc sulphate. W.G.C.

Modified Viscose Rayon Yarns

DuP *BP* 741,728

Yarns having high tenacity, substantially non-crenulated surfaces and a high proportion of skin are produced by adding to the viscose 0·2–2% of a compound of general formula $R^1O(CH_2CH_2O)_n{\cdot}R^2$ (R^1 = Alk or Ar; n = 1–4; R^2 = H, Alk or Ar) e.g. phenoxyethanol. W.G.C.

Electrically Conducting Viscose Rayon

Pirelli *BP* 743,909

Incorporation of carbon black into viscose (3–100 vol. of pigment per 100 vol. of dry cellulose fibre obtainable from the viscose) results in fibres produced from the mixture being electrically conductive. C.O.C.

Dextran Ether Fibres

Commonwealth Engineering Co. *USP* 2,702,231

A dextran ether and a cellulose nitrate lacquer are mixed with a mutual solvent to form a viscous mass. This is extruded through spinnerets to produce fibres. C.O.C.

Regenerated Protein Fibres

ICI *BP* 742,001

Regenerated protein fibres of improved colour are obtained if an alkali metal fluoride is added to the spinning bath or subsequent treating liquors. W.G.C.

Regenerated Protein Fibres

American Patents Corpn. *BP* 743,576

Fibres of increased strength are obtained by pretreating the protein with a nitrogenous substance, e.g. urea, and one or more fatty substances of animal origin, e.g. whale oil. W.G.C.

Esterifying Casein Fibres

Courtaulds *BP* 741,740

Esterification of casein fibres is carried out in a J-box fitted with a conveyor belt to prevent undue compression of the fibres at the bend of the J. W.G.C.

Water-soluble Fibres from Polyvinyl Alcohol Copolymers

Kurashiki Rayon *BP* 743,165

The solubility in water of polyvinyl alcohol fibres is improved by using a copolymer containing 2–10 mol. % of allyl alcohol. W.G.C.

Matt Acrylonitrile Fibres

P. Halbig *BP* 741,847

In the wet spinning of polyacrylonitrile from nitric acid solution, matt fibres are obtained when the coagulating bath is at 25–50°c. W.G.C.

Continuous Blending of Pigments in Spinning Dopes (I p. 109)
Effect of Nitrogen Oxide Gases on Processed Cellulose Acetate Film (IX p. 124)
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Electron-microscopic Observation of Monolayers of Synthetic Linear Polymers (XIII p. 129)
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Measurement of the Extent of Delustring of Filament Fabrics. II—Reflection of Unpolarised Light (XIV p. 133)
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Electrical Resistance of Synthetic and Cellulose Acetate Fibres (XIV p. 133)

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

Soiling of Natural and Manufactured Fibres from Aqueous Systems

AATCC New York Section

Amer. Dyestuff Rep., **44**, P 815–P 826 (21 Nov. 1955)

Fifteen natural and man-made fibre fabrics are soiled with two representative soils—vacuum-cleaner soil and synthetic—which are applied by padding, spraying, and immersion techniques, these being described. Four different standard washing methods are then applied, and the results assessed by reflectance measurements. In most instances soil applied by spraying produces the heaviest soiling and is the most difficult to remove. Padding produces low adhesion and easy removal, and immersion slightly heavier and slightly more retentive soiling. Types of fibre and physical construction have a great influence, and spun yarns in general pick up and retain more soil. Fabrics are listed in the following order of retention, the worst first: wool (Lanaset treated) > spun Orlon staple shirting > spun viscose rayon staple gabardine with acetate rayon warp, viscose rayon weft > filament Dacron shirting > spun nylon staple blouse > cotton Indian Head > spun viscose rayon gabardine, no finish > cotton broadcloth > filament nylon shirting > spun Dacron shirting > silk crêpe > bright filament acetate rayon taffeta > spun viscose gabardine, resin finish > filament Orlon shirting > two-bar nylon tricot. It is considered that standardised laboratory methods can be used to forecast behaviour in actual use. J.W.B.

Electron-microscopic Study of the Effect of Cationic Wetting Agents on Aqueous Stearic Acid Sol

R. S. Roy and D. L. Bhattacharya

J. Indian Inst. Science, **A 37**, 254–257 (Oct. 1955)

Electron micrographs show that, when colloidal stearic acid is mixed with a solution of *N*-butylguanidine sulphate at room temp., the elongated laminar particles are practically unchanged in shape; but when it is mixed with solutions of triethanolamine hydrochloride and cetylpyridinium bromide, the shape of the particles is changed rapidly and there is a tendency towards coagulation. At the boil, colloidal stearic acid particles become spherical, but revert to laminar shape on cooling. Treatment with the cationic agents makes this change irreversible, and the particles remain spherical. W.K.R.

Fastness to Light and Washing of some Fluorescent Brightening Agents

W. Markiewiczowa

Zesz. Nauk. Politech. Łódz., No. 2 (*Chemia* 1), 69–76 (1954)

Eleven fluorescent brightening agents, eight of which are commercial products, were examined. All showed a light fastness of 1 in the Fade-Ometer. Those compounds which contained dehydrothio-*p*-toluidine-*o*-sulphonic acid in the molecule, and also Uvitex RS (Ciba), lost all their fluorescent properties after 5 hr. exposure. Fastness to washing varies between 1 and 4. The highest fastness was exhibited by "Thiazole 4" and Blancophor B. Fixing Agent W improves fastness to washing by 0·5 degree without affecting light fastness. T.Z.W.

Relation between the Degree of Polymerisation and the Mechanical Properties of Cotton Fibres degraded by Hypochlorite
A. Parisot and A. Fresco
Bull. Inst. Text. France, (56), 7–23 (Oct. 1955)

Cotton fibres were subjected to repeated treatments each consisting in (*a*) washing in alkaline soap solution, (*b*) bleaching in alkaline hypochlorite, (*c*) dechlorination in dilute sodium bisulphite solution, and (*d*) drying. The dry and wet strengths of the fibres and the D.P. (measured viscosimetrically) were found to decrease as the number of treatments increased. The rate of degradation depended on the concentration of hypochlorite. At low hypochlorite concentrations the degradation followed a linear law; at higher concentrations the relationship was more complex. The wet strength decreased more rapidly than the dry strength, and became lower than the latter when the D.P. fell to 600–700. It is suggested that this last result is due to the occurrence in the polymer chains of regions which are more accessible to reagents than is the rest of the chain. J.C.F.

Hypochlorite Bleaching
K. E. Lekander and L. Stockman
Svensk Papperstidning, **58**, 775–785 (15 Nov. 1955)

The influence of pH, temperature, and pulp consistency during hypochlorite bleaching on the properties of chlorine-bleached, alkali-treated sulphite pulp has been investigated. On raising the pH from 7 to 11, brightness, viscosity, and α-cellulose and carboxyl-group contents increased, while β-cellulose, Cu No., and yellowing decreased. The effect of temperature up to 60°C. was negligible, except that the reaction rate was doubled for a rise of 7°C. Pulp consistency seemed to affect only brightness, which decreased on bleaching at high consistencies, even when the chemicals and fibres were perfectly mixed. K.W.

PATENT

Bleaching Polyacrylonitrile Fibres
Union des Fabriques Belges de Textiles Artificiels "Fabelta"
BP 742,349

Polyacrylonitrile fibres are bleached to a brilliant and stable white by treating them at 20–100°C. with an aqueous permanganate at pH < 7 and then with an aqueous reducing agent. Thus polyacrylonitrile fibre (1 kg.) is immersed for 10 min. at 60–100°C. in 40 litres of 0·1% aq. $KMnO_4$ brought to pH 2 with H_2SO_4. It is then dried without heating and treated for 15 min. in boiling 2% aq. $NaHSO_3$ brought to pH 2 with H_2SO_4. Finally it is rinsed and dried in absence of heat. C.O.C.

Effect of Micellar Size on Physicochemical Properties of Surface-active Agents (III p. 109)

American Bleaching and Dyeing Practice (VIII below)

Measuring the Whitening Effects of Fluorescent Brightening Agents and Perborate Bleaches on Cotton (XIV p. 134)

VIII—DYEING

American Bleaching and Dyeing Practice
J. K. Skelly
J.S.D.C., **72**, 7–15 (Jan. 1956)

The applications of the established American bleaching and dyeing processes for cotton piece goods are discussed. An outline is given of the Becco, du Pont, and Mathieson continuous bleaching systems. The practical uses of the Williams unit and pad–steam dyeing ranges are indicated. Particular attention is paid to the factors influencing the advantages and limitations of each of these processes. AUTHOR

Dyeing Wool with Direct Dyes
AATCC Philadelphia Section
Amer. Dyestuff Rep., **44**, P 806–P 814 (21 Nov. 1955)

Approx. 260 direct dyes are applied to wool. Rate of exhaustion can be controlled by adjustment of pH, and all dyes will exhaust at pH 6; the rate is significantly lower and more uniform than with comparable acid and milling dyes at the same pH. Fading tests show that these dyes cover a complete fastness range, and some are very fast; troubles in washing due to staining of cotton are greater than with milling dyes, but can be largely overcome by aftertreatment with chromium fluoride. Auxiliaries can also be used to control tippiness. Although these dyes are generally avoided because of their "direct" classification, it is thought that some are worthy of serious consideration for wool dyeing. J.W.B.

Application of Acid and Metal-complex Dyes to Weighted Silk
G. Dierkes and K. Rooseboom
Z. ges. Textilind., **57**, 1504–1505 (20 Nov.–5 Dec. 1955)

Degumming followed by weighting of silk piece goods is applicable to 100%-silk materials only and not to mixtures. Tram must not be present, and the pieces should not be tightly woven. The production of level dyeings fast to light and washing is not too easy on degummed and weighted material. Acid dyes are generally used, as they are the easiest to apply, but they may not always give the required fastness. Palatine Fast (BASF) dyes are not suitable for weighted silk, as the high acidity of the dyebath may remove the weighting. The newer metal-complex dyes are of interest, however, as they are applicable from a neutral or weakly acid dyebath and give good fastness. Careful temp. control and the addition of products such as Uniperol W (BASF) or Avolan IS (FBy) are advisable to promote level dyeing. Dyeing should be carried out with ammonium acetate followed by acetic acid if needed to ensure minimum removal of weighting. Good wash fastness and a light fastness of 5–6 are obtained. B.K.

Dyeing of Synthetic Fibres. III—Adsorption of Sulphur Dyes by Vinylon
Y. Hamasaki and S. Wakimoto
Science and Ind. (Japan), **29**, 222–225 (1955)
Chem. Abs., **49**, 16440 (25 Nov. 1955)

When dyeing Vinylon cloth (acetylated 38·1%) with Sulphur Black B, addition of up to 10% of Na_2S increased adsorption of dye as did addition of up to 2–3% of NaHS and a small amount of $Na_2S_2O_4$. In most cases deeper dyeings were obtained with NaHS than with Na_2S. Treatment with $K_2Cr_2O_7$, $CuSO_4$ or acetic acid after dyeing improved fastness to light and washing but not to rubbing. C.O.C.

Dyeing and Finishing of Nylon Ribbons
W. S. Willson
J.S.D.C., **72**, 15–18 (Jan. 1956)

The process of setting nylon fabrics is explained and discussed, with particular reference to plant and methods necessary for nylon ribbons. The best sequence of operations for dyeing and finishing is suggested with emphasis on the precautions necessary to achieve good results. Alternative methods for future development are referred to, with suggestions for quicker or more direct finishing processes. Selection of dyes and bleaching agents, and methods of producing durable effects in finishing, are discussed. AUTHOR

Use of Carriers in the Dyeing of Polyester Fibres
G. Dierkes and G. Römer
Melliand Textilber., **36**, 1170–1174 (Nov.), 1271–1275 (Dec. 1955)

The actions of the following were investigated: *o*- and *p*-phenylphenol, 2:2′-dihydroxydiphenyl, 4:4′-dihydroxydiphenyl, *o*- and *p*-*cyclo*hexylphenol; methyl phenyl and ethyl phenyl ethers, diphenyl ether, phenoxyethanol; benzyl and phenylethyl alcohols, *cyclo*hexanol; benzophenone, *cyclo*hexanone; phenyl-methyl-aniline. The use in combination of the diphenyl, phenyl, and *cyclo*hexyl compounds is discussed. The use of carriers with diammonium phosphate is described, and desiderata for an ideal carrier are enumerated, concluding with a reference to Resolin (FBy) dyes, which require no carrier. S.R.C.

Dyeing Vinylon black with Oxidation Dyes. IV—Effect of Combined Oxidation and the Damage to the Fibres
Y. Urahata and Y. Hamasaki
Science & Industry (*Japan*), **29**, 219–221 (1955)
Chem. Abs., **49**, 16440 (25 Nov. 1955)

Chlorite-bleached Vinylon when dyed black with *p*-$NH_2C_6H_4NHC_6H_4HCl$ (I) together with aniline, (CH_2O)-(H_2N)-$C_6H_2)_2$, $(p\text{-}NH_2C_6H_4)_2NH$, p-$(NH_2)_2C_6H_4$ or 1-

naphthylamine hydrochloride gave reflection curves in which the dominant wavelength shifted more towards the complementary colour with lowering of purity than when dyed with I alone. The oxidation damage caused to Vinylon fibres by this method of dyeing is only half that done to cotton and only one fourth that done to regenerated cellulose staple fibre. C.O.C.

Dyeing of Velours
C. Alabouvette and C. Rouanet
Bull. Assocn. Française Chim. Ind. Cuir, **17**, 206–218 (Nov. 1955)

In the dyeing of velour leathers two opposing factors must be reconciled— the penetration of the dye, and its aggregation. This is easy with pale colours, and a wide range of colouring matters is available; metal-complex dyes are preferable. For the production of medium depths it is best to use dyes, specially selected for the purpose, which are intermediate in type between those which penetrate freely and those which are substantive; penetration is secured in mildly ammoniacal soln., and good fastness to wet rubbing results from subsequent acidification. Deep colours are obtained with the same dyes, to secure penetration, and a further treatment with basic dyes in the presence of non-ionic or, better, cationic agents. With blacks this method does not give dyeings of good fastness to wet rubbing and the leather is often reduced in tear-strength. A better method for blacks is to use a navy blue or violet component of good penetration; this is diazotised and developed with a diamine (*m*-tolidine) *in situ*. This increases not only the number of chromophoric and auxochromic groups, but also the mol. wt., and thus yields dyeings of good fastness to light and to wet rubbing. The various classes of dye are discussed, in arriving at these conclusions, with particular regard to their interaction with leather, and to the effect of pretreatments such as tannage and dressing. J.W.D.

PATENT

Dyeing Viscose Rayon Cakes
ICI *BP* 743,660

Level dyeings are obtained with a shorter dyeing time if the ratio of dye liquor to the weight of the cakes is not more than about 8 : 1. C.O.C.

Dye Constitution and Dischargeability— Subsequent Development of Discoloration on Polyamide Fabrics (IX below)

IX— PRINTING

Dye Constitution and Dischargeability— Subsequent Development of Discoloration on Polyamide Fabrics
B. Kramer
Melliand Textilber., **36**, 1295–1300 (Dec. 1955)

The amine residues from reduction are more difficult to remove from polyamide fabrics than from cellulose, and can turn yellow or brown with atmospheric oxygen. Successful discharging depends on the constitution and the purity of the dye, on the substrate, the steaming conditions, and the nature of the discharge products. A number of disperse, acid, and direct cotton dyes are considered. S.R.C.

Recent Progress in the Diazotype Process. III— Photodiazotyping on Wool and Silk
L. Mester
Science et Inds. phot., **26**, 354–356 (1955)
Chem. Abs., **49**, 15577 (25 Nov. 1955)

The bright yellow of poor light fastness obtained on wool or silk by treatment with HNO_2 is almost entirely caused by diazotisation of the tyrosine component. Introduction of a nitroso group into the protein molecule occurs only to a slight extent and the product has excellent fastness to light. The absorption spectrum of HNO_2-treated wool (I) or silk (II) has the same absorption maximum and form as 1-diazo-2-naphthol-4-sulphonic acid (III). Samples of I and II that have been exposed to light yield distinctly different colours than do unexposed samples when treated with alkaline β-naphthol, a neutral salt of H-acid, or solutions of phloroglucinol (IV) or resorcinol (V) in presence of $NiSO_4$ and developed with NH_3. Exposed samples become deep violet when coupled with a diazo compound in presence of NH_3 whereas unexposed samples remain almost unchanged. Photolysis of many diazo compounds yields phenols capable of coupling with diazo compounds. The coupling activity of I and II towards IV, V and Na-β-naphthol-3:6-disulphonate are compared with that of diazotyrosine (VI), III, *p*-diazodiphenylamine and *p*-diazodimethylaniline. The coupling activity of I and II was similar to that of III and VI. The dye formed by coupling I and II with IV was extracted from the samples with 10% NaOH, it had the same ultraviolet absorption spectrum as the dye from VI and IV. Continuous tone images can be formed by exposing I and II through a negative. C.O.C.

Effect of Nitrogen Oxide Gases on Processed Cellulose Acetate Film
J. F. Carroll and J. M. Calhoun
J. Soc. Motion Picture Television Engrs., **64**, 501–507 (1955)
Chem. Abs., **49**, 15578 (25 Nov. 1955)

N_2O and NO are harmless but NO_2 even at low concentration is a serious danger to safety film. The order in which the film components are attacked is (1) Ag or dye image, (2) gelatin, (3) acetate base. The rate of attack is increased by increase in R.H. C.O.C.

PATENTS

Printing on Fabric of Glass Fibres
Owens-Corning Fiberglas Corpn. *BP* 742,073

The printing paste contains an organic pigment, a butadiene-acrylonitrile copolymer (e.g. Hycar OR-25), a water-soluble cellulose ester or ether, and a salt of a starch derivative containing carboxylic and sulphuric acids groups and which can be insolubilised by treatment with a Werner complex compound in which a trivalent nuclear Cr atom is co-ordinated with an acrylic carboxylic acid group of 6 C and an aldehyde. The printed fabric is dried at high temperature and then treated with an aldehyde and a Werner complex of the above type. A suitable starch derivative is Clearflo H (National Starch Products Co.). C.O.C.

Printing on Coated Vapour-proof Regenerated Cellulose Film
Transparent Package Co. *USP* 2,702,254

Film of the type described in *USP* 2,627,483 can be printed if it is treated with a solvent, e.g. xylol, nitrobenzene or ethyl methyl ketone, under carefully controlled conditions, and the residual solvent removed before the film is printed. C.O.C.

Prints on Rubber
G. Ingram *BP* 735,780

A solution of rubber, factice and vulcanising agents is applied to the surface of the semi-vulcanised rubber article and to the surface of the desired print (in reverse) in ordinary printers' ink on paper. The two partially dried surfaces are pressed together and the paper removed after soaking with an alkaline soft soap solution. The print is protected with a film deposited from compounded rubber latex and the whole is vulcanised by heating. E.C.

Sensitising Paper to Application of Colour-reactants
National Cash Register Co. *USP* 2,702,765

Paper is impregnated with particles of an acidic metal silicate by passing it successively through two liquors which react to precipitate the silicate in the paper. One of the liquors contains a little of a latex binder. The impregnated paper forms a distinctive colour when printed or written on with a colour reactant. Thus paper impregnated with magnesium silicates becomes deep blue when a colourless solution of Crystal Violet lactone dissolved in liquid chlorinated diphenyl is applied to it. C.O.C.

Multilayer Colour-photographic Positive Material
Photo-Chemical Co. *BP* 743,759

Coloured Layers of Polyvinyl Materials (XIII p. 130)

X—SIZING AND FINISHING

Effect of Mercerisation on the Properties of a Crease-resistant Cotton Fabric
AATCC Rhode Island Section
Amer. Dyestuff Rep., 44, P 779–P 785, P 790 (7 Nov. 1955)

The effect of varying degrees of mercerisation on the crush resistance and other related properties of a typical cotton fabric when resinated is studied on the plant scale. The cloth is bleached by continuous peroxide, and by kier boil followed by chemic. Portions are then mercerised with six different strengths of NaOH and double-mercerised with two strengths. A urea–formaldehyde paste resin, and a methylated melamine syrup resin, are applied. Crease and abrasion resistance and tensile and tear strengths are measured at various stages of processing, and all the processes are described in detail. Loss in tensile strength and in tear strength, which normally results from resination, is materially reduced by premercerisation, and further improvement is effected by adding softener either to the padding liquor or as an aftertreatment. The optimum mercerising strength is 45–65°Tw. There is no effect on crease recovery, abrasion resistance, or shrinkage control. There is a definite correlation between the efficiency of mercerisation as measured by Ba number, dye adsorption, and dyeing tests and the improvements in physical properties. J.W.B.

Effect of Additives to Thermosetting Resin Baths on the Physical Properties of the Treated Fabric
T. F. Garvey
Amer. Dyestuff Rep., 44, P 791–P 794 (7 Nov. 1955)

Changes are described in physical properties of resin-treated fabrics resulting from the addition of elastomers, thermoplastic and thermosetting resins, repellents and softeners to a normal thermosetting resin finishing bath. Wrinkle recovery, degree of resilience, tear strength, and abrasion resistance may all be improved by selected additives, though some operate at extremely critical optimum concentrations. J.W.B.

Delustring and Imparting a Rough Finish to Steelon (6-Nylon)
A. Nowakowski, S. Chrzczonowicz, and L. Brajter
Zesz. Nauk. Politech. Łódz., No. 9 (*Chemia* 3), 73–79 (1955)

Continuous-filament Steelon yarn is immersed for 30 min. in formic acid (30%) at 20°c., hydroextracted, and entered at 75°c. for 2 min. into a bath containing 4% of the *N*-hydroxymethyl derivative of steelon and 1% $NaHCO_3$ in 87% ethanol. It is then hydroextracted and soured for 1–2 min. in 5% HCl, washed well, and dried. The finished yarn increased in weight by 5%, increased slightly in strength, and dyed deeper colours. The effect produced is fast to washing. T.Z.W.

Improving Steelon (6-Nylon) Fibres
S. Chrzczonowicz
Zesz. Nauk. Politech. Łódz., No. 9 (*Chemia* 3), 81–91 (1955)

A matt finish is produced on Steelon fibres by immersion in a solution containing 0·5–2·0 g. Steelon and 18 g. calcium chloride in 100 ml. of 96% ethanol at 20 ± 5°c. for 20 min. Impregnation is followed by— (*a*) washing in water until free from chloride ions; (*b*) passing through 2% barium chloride solution followed by treatment in 2% sodium sulphate solution and finally washing in water; (*c*) passing through 2% ammonium fluoride solution and washing with water. Different degrees of matt appearance can be produced by modification of the composition of the main bath within the limits stated. The mechanical strength of the fibre is not affected if ethanol is used as a solvent in the preparation of the main bath. T.Z.W.

PATENTS

Permanently Crimping Superpolyamide Threads
Cotonificio di Lombardia S.A. Industriale *BP* 742,352

The thread is given a twist of ⩽3000 turns per metre, wound on a rigid support, treated with saturated steam at 110–115°c. for 180–210 min. and finally untwisted. Steaming is preferably done in a vacuum of about 74 cm. Hg.

The crimped thread can be dyed or ironed at 130–140°c. without deterioration. C.O.C.

Size
Atlas Powder Co. *USP* 2,702,796

A size which is readily dispersible in water consists of polymethacrylic acid, a plasticiser, and a lubricant. The size is applied at pH ⩽4·5–6·0. Such a mixture gives good sizing and is non-corrosive. C.O.C.

Dry Sizing
Gebrüder Sucker *BP* 742,308

Threads or batches of threads are dry-sized by passing over one or more rollers, which may have a peripheral speed greater than that of the fibres, and which may be hollow with a perforated surface fitted with a brush-like or plush cover, the size being supplied through the inside. The contact angle of the threads to the rollers can be varied to suit the degree of mechanical action and thus the size penetration. Heat regulators, e.g. concave inf.-red reflectors, may be provided adjacent to the rollers to control size fluidity and thus the amount applied. J.W.B.

Modifying Cellulose Fibres with Alkali Solutions of Copper or Nickel Biuret
Hathaway Manufacturing Co. *USP* 2,702,228

Aqueous solutions containing biuret, a copper or nickel compound and an alkali metal hydroxide are swelling agents and solvents for cellulose. Used on cotton marquisettes, voiles, etc. they remove projection fuzz, impart lustre and a stiff handle and confer improved dimensional stability when washed. C.O.C.

Chlorinating Wool
Scholler Brothers *USP* 2,702,737

Wool containing 0·3–2·0 milliequivalents of acid per g. of air-dry clean fibre, is treated for 2 sec.–10 min. with an aqueous solution of a chlorinating agent while maintaining the solution at pH 7·2–11·5, the acidity in the wool being retained throughout chlorination. This enables closer control of chlorination. C.O.C.

Resinous Compositions containing Hydrous Oxides for Reducing the Felting Properties of Wool
American Cyanamid Co. *USP* 2,702,258

When using alkylated methylol melamines for reducing the felting power of wool addition of a water-insoluble metallic hydrous oxide increases the efficiency of the treatment. Thus methylated trimethylol resin (45 parts by wt.) is dissolved in water (200). To this solution is added a dispersion formed by dissolving 1·3 parts of an 88/12 mixture of diammonium hydrogen phosphate and hexamethylenetetramine in water (100) and adding $CoSO_4.7H_2O$ (1·75). Wool impregnated with its own weight of the resulting mixture, dried at 80°c. and baked for 9 min. at 140°c. has less residual shrinkage than wool similarly treated in absence of $CoSO_4$. C.O.C.

Adhesives for Cellulose Acetate and Bonding of Weftless Ribbons
BrC *BP* 741,992

Weftless ribbons are made by bonding a narrow warp of cellulose acetate yarns, or yarns of cellulose acetate together with cellulosic yarns, with a solution of a polymer which is capable of forming flexible films and which will dissolve in an aqueous liquid which is not a solvent or strong swelling agent for acetate rayon, the solution also containing diethylene glycol diacetate in suitable plasticising proportions. In one example coloured yarn is coated with its own wt. of a solution comprising polyvinyl alcohol (100), diethylene glycol diacetate (80), water (720), and matched cotton dye (0·5) at 60°c. After drying at 70°c. the ribbon is calendered; ornamental effects may be embossed if desired. J.W.B.

Resin-coated Nylon Fabric of High Tear Strength
Fabric Research Laboratories *USP* 2,702,764
If nylon fabric before being coated with natural rubber or a polyvinyl resin is impregnated with a lubricant, e.g. a plasticiser for synthetic or natural resins, then the tear resistance and service life of the coated fabric is markedly enhanced. C.O.C.

Water-repellent and/or Gas-fume-fading-resistant and/or Flame-resistant Finish
Phillips Petroleum Co. *USP* 2,702,763
Fabric, made from any type of fibre, is impregnated with a polymer of a pyridine derivative containing at least one vinyl group. Thus cotton, rayon and wool fabrics impregnated with an aqueous acetic acid solution of a 50/50 butadiene/2-methyl-5-vinyl pyridine copolymer or of poly-2-methyl-5-vinyl pyridine and dried were given a water repellent finish, resistant to dry-cleaning and laundering. Blue acetate fabric similarly impregnated with poly-2-methyl-5-vinyl pyridine was rendered resistant to gas fume fading. C.O.C.

Synthetic Resinous Coatings for rendering Fabrics and Leather Water-impermeable but Vapour-permeable
Kodak *BP* 742,279
A composition containing an ester of formula CH_2:CR^1-$COOR^2$ (R^1 = H or Alk of 1–4; R^2 = Alk of 1–6 C) polymerised at 45–120°C. with a polymer of formula

$$-(CHOH{\cdot}CH_2)_x(CH(CH_2){\cdot}O{\cdot}CO{\cdot}R^3)_{100-x}$$

(R^3 = Alk; x = percentage of groups based on vinyl alcohol) yields a softened and flexible film. It is suitable for producing waterproof coatings on cloth or leather intended for garments. C.O.C.

Infrared-reflecting Fabric
Minnesota Mining & Manufacturing Co. *USP* 2,703,772
Metal is vapour-coated on to a smooth-surfaced carrier film. The metal surface is coated with a highly flexible curable adhesive and then applied to a fabric followed by curing and removing the film backing. This leaves the fabric with a bright and highly reflective metallic coating. C.O.C.

Glass Fabric resembling Leather
Corning Fiberglas Corpn. *USP* 2,703,774
Glass fibres are blended with a small proportion of a synthetic organic fibre which can be shrunk by heat or solvents, e.g. Vinyon. The blend is made up into a thick knitted fabric and then heat and/or solvent treated to cause the organic fibres to shrink. This yields a fabric which has considerable stretch in any direction without imposing any strain on the glass fibres. Impregnating the fabric, during or after shrinking, with a resin or resin-forming solution yields a product having leather-like properties. C.O.C.

Preparation of an *N*-Hydroxymethyl Derivative of Steelon (6-Nylon) soluble in Methanol and Ethanol (III p. 110)
Dyeing and Finishing of Nylon Ribbons (VIII p. 123)
Comparative Effects of some Fungicidal Substances upon Fungi occurring in Wet Pulp (XI below)
Biological Activity of Phenylmercuric Acetate in Presence of Groundwood Pulp Fibres (XI this page)
Continuously Depositing Gelatin in or on Travelling Strip Material (XI p. 127)
Burning Behaviour of some Cellulose Ester Film Compositions (XIII p. 128)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Comparative Effects of some Fungicidal Substances upon Fungi occurring in Wet Pulp
S. K. L. Freyschuss
Svensk Papperstidning, **58**, 815–817 (30 Nov. 1955)
Phenylmercuric acetate and 8-hydroxyquinoline (oxine) were found to be particularly effective *in vitro*, whereas a ten times as high concentration is required for sodium pentachlorophenoxide and 3:5-dimethyltetrahydro-1:3:5:2H-thiadiazine-2-thione to achieve the same effect. K.W.

Biological Activity of Phenylmercuric Acetate in Presence of Groundwood Pulp Fibres
S. K. L. Freyschuss
Svensk Papperstidning, **58**, 755–757 (31 Oct. 1955)
The biological activity of phenylmercuric acetate is markedly reduced in the presence of groundwood pulp at concentrations inhibiting fungal growth *in vitro*. Addition of NaCl or KCl increases the biological activity, whereas Na_2SO_4 and $Al_2(SO_4)_3$ have practically no effect. K.W.

Effect of Grinding on the Electrokinetic Properties of Sulphite Cellulose
V. I. Yur'ev, S. S. Pozin, and L. N. Bilich
J. Appl. Chem. U.S.S.R., **28**, 1131–1134 (Oct. 1955)
The electrokinetic potential (determined by the streaming-potential method) of sulphite cellulose in contact with water or 0·0001 N-KCl is reduced numerically when the cellulose is ground. For bleached demineralised sulphite cellulose in its original state and after very thorough grinding, the potentials are, respectively, −7·4 mv. and −1·6 mv. against water and −9·3 mv. and −3·1 mv. against 0·0001 N-KCl. This effect of grinding is to be attributed to increase in the adsorptive power of the cellulose and to increase in the extent of electroneutral hydration, which results from the freeing of polar groups by the rupture of hydrogen bridges between macromolecules. Grinding results also in increased surface conductivity. A.E.S.

Interpretation and Measurement of Contact Angles of Water on Paper
E. Back and B. Lundin
Svensk Papperstidning, **58**, 758–763 (31 Oct. 1955)
A review of recent theoretical and experimental work on contact angles on rough or non-homogeneous surfaces consisting of different chemical components is followed by a discussion of contact angles of water on paper surfaces. Sized paper is regarded as having a rough surface built up of interspersed areas of hydrophobic rosin or Al resinates and of hydrophilic and water-absorbent cellulose. Although contact angles are difficult to interpret, apparent contact angles represent a defined surface property provided that the paper surface is sufficiently sized to permit water absorption to be neglected during the constant period of 10 sec. necessary for measurement. An improved apparatus and details of measurement are described, and data given showing the increasing standard deviation with decreasing apparent contact angle. K.W.

Relationship between Dielectric Constant and Water Vapour Accessibility of Cellulose
D. E. Kane
J. Polymer Sci., **18**, 405–410 (Nov. 1955)
In the accessibility range 35–85% the relationship between dielectric constant and water vapour accessibility is very nearly linear. Extrapolation of the relationship suggests that the dielectric constant of accessible cellulose is about 9, that of inaccessible cellulose being about 4. The dielectric constant of the cellulose can be regarded as that of a two-component system, the two components being the accessible and inaccessible fractions. W.R.M.

Comparative Study of the Oxidation Reactions of Pectic Substances, of Cellulose, and of the Structural Units of these Substances
O. P. Golova and N. S. Mayat
II—Oxidative Degradation of Sodium Polygalacturonate by means of Molecular Oxygen, Sodium Metaperiodate, and Hydrogen Peroxide
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 899–905 (Sept.–Oct. 1955)
Polygalacturonic acid (purified hydrolysate of citrous pectin) is treated with oxidising agents (O_2, $NaIO_4$, H_2O_2) in alkaline soln. (pH 12–13), and some of the changes occurring are followed by viscosity determinations on 0·5% soln. in 0·05 N-NaOH and by aldehyde and carboxyl estimations. It is considered that the degradation indicated by the viscosity changes is to be attributed mainly to the formation of new functional groups at C-2 and C-3, which induce rupture at the glycoside linkages in an alkaline medium. These reactions are effected not only by the specific oxidant $NaIO_4$, but also by O_2 and H_2O_2,

which, however, react at an appreciable rate only at ~100°C., although H_2O_2 reacts rapidly at room temp. when a catalyst is present.

III—Reactivities of the Structural Units of Cellulose and Pectic Substances to Oxidation by Sodium Metaperiodate and Hydrogen Peroxide

Ibid., 1078–1084 (Nov.–Dec.)

The oxidation of methyl α-galactosiduronic acid (I; cf. polygalacturonic acid) and of methyl α-glucoside (II; cf. cellulose) with $NaIO_4$ and with H_2O_2 in acid (pH 4·2) and in alkaline (pH 11–12) soln. is followed by polarimetric measurements and by the determination of formic acid formed by Malaprade-type oxidation. I is more readily oxidised than II. Comparison of the oxidation of I with that of methyl α-galactoside (III) (effect of replacement of CH_2OH by COOH) and of the oxidation of II with that of III (effect of change of configuration at C-4) shows that the difference in reactivity between I and II—and presumably, therefore, between polygalacturonic acid and cellulose—is to be attributed to difference in configuration at C-4. A.E.S.

PATENTS

Stabilised Cellulose Ether Compositions

Hercules Powder Co. *BP* 742,659

Thermoplastic cellulose ethers can be stabilised against viscosity degradation and discoloration by adding or incorporating with the cellulose ether (ethylcellulose or deashed ethylcellulose) a small amount of improved phenolic stabilisers (0·25–6·09%) of the polycyclic type having at least two non-condensed aromatic nuclei joined by methylene groups and each nucleus having a hydroxy group in *ortho* position or adjacent to the C atom attached to the methylene group. K.W.

Photosensitive Paper-base Material

Adelphi Research & Manufacturing Co. *BP* 742,444

The coating material consists of a fluid aq. dispersion of a basic Al carbonate gel prepared by neutralising an acid Al salt with an alkaline carbonate such that the gel will contain at least 0·6 to 1·6 mol. of CO_2 per mol. of Al_2O_3, and the pH of the aq. suspension containing the equivalent of about 4% Al_2O_3 will be about 6·0–7·5. For the coating process, which may be conventional brush, spray or roll coating, the gel is diluted preferably to between 0·5 and 2% Al_2O_3 at pH 5·5–7·0. This base material when sensitised particularly with blue-print solution results in a more intense colour, has a greater shelf-life, and retains its strength and dimensional properties to a large extent. K.W.

Continuously Depositing Gelatin in or on Travelling Strip Material

J. Dungler *BP* 741,351

A method of sizing or coating continuous strip, e.g. a paper web, at open width with a solution of gelatin followed by rapidly drying the treated web in a thermally insulated chamber in which air is replaced by steam and jets of superheated steam or vapour evaporate the solvent maintained at b.p. K.W.

Sensitising Paper to Application of Colour-reactants (IX p. 124)

Influence of Oxygen on the Cuprammonium Viscosity of Cellulose (XIV p. 134)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Structure of Collagen

A. Rich and F. H. C. Crick

Nature, **176**, 915–916 (12 Nov. 1955)

The structure proposed by Ramachandran and Kartha (*ibid.*, **176**, 593 (1955)) is criticised. (1) The structure is stereochemically unsatisfactory. In particular, there is a very short C_β–C_β contact of 3·3 A. (normally 3·6–4·0 A.), and an extremely short C_β–O contact of 2·6 A. (normally 3·2–3·5 A.). Further, the hydrogen-bond angles are beyond the limits usually found. (2) It is not compatible with recent work, which shows that—

–glycine–proline–hydroxyproline– (I)

is a common sequence in collagen. Optical diffraction data indicate that the proposed structure is broadly correct, but incorrect in detail. By taking a compact group of three adjacent chains from the polyglycine lattice (space group $P3_1$), and twisting them as in the Ramachandran–Kartha structure, to form a similar coiled coil, two different structures may be obtained; both have a right-handed major helix and a left-handed minor helix, arranged to fit the observed non-integer screw, and both will accommodate the sequence I. Evidence is discussed supporting the contention that one of these two structures is correct—the authors are as yet unable to decide which. In either event, collagen appears to be a three-chain, coiled-coil structure, with one set of systematic hydrogen bonds. J.W.D.

Apparent Transformation of Collagen Fibrils into "Elastin"

D. Burton, D. A. Hall, M. K. Keech, R. Reed, H. Saxl, R. E. Tunbridge, and M. J. Wood

Nature, **176**, 966–969 (19 Nov. 1955)

Electron-microscopical, biochemical, and histological studies are reported; these indicate that under conditions which lead to the transformation of collagen fibrils into structures closely resembling those found in naturally occurring elastic tissue, protein fractions rich in hydroxyproline (I) and arginine (II) are released from the collagen. A deep-seated and complex breakdown of some of the polypeptide chains of collagen is involved, and protein fragments low in I and II are simultaneously liberated. The degree of degradation, and the composition of the resultant amorphous elastomuoin, can vary widely without affecting the ill-defined morphology of the elastin fibre. J.W.D.

Concept of Olation

A. Küntzel

J. Soc. Leather Trades Chem., **39**, 359–368 (Nov. 1955)

In criticising the work of Shuttleworth (*ibid.*, **39**, 123 (1955)), the author points out that basicity should be defined as the number of OH groups to Cr, expressed as % of equivalents of Cr, and olation as the % of OH groups that exist as olation bridges. Olation refers to the ageing process of Cr cpd. after addition of NaOH, characterised by (1) development of acid-resistance, (2) decomp. into products of smaller particle-size, and (3) fall in pH value. This concept is restricted to sol. basic Cr salts, which are related to two different types of insol. $Cr(OH)_3$. The non-olated basic salts are the precursors of α-$Cr(OH)_3$, and the olated basic salts are the precursors of the β-form. These types, and their constitution, are discussed. J.W.D.

Use of Urea–Formaldehyde Resins in Tanning

P. Chambard

Bull. Assocn. Française Chim. Ind. Cuir, **17**, 199–205 (Nov. 1955)

The optimum conditions for the tannage of suède leather with urea–formaldehyde resins (UF) have been studied. Satisfactory results can be obtained only with bishydroxymethylurea, and this should be formed by the application of urea and formaldehyde amounting to ca. 10% on the weight of the leather. The acid used to promote polymerisation and its concentration affect the end-result; H_2SO_4 or (COOH)$_2$ is preferred, in concn. of ≯10%, and the optimum pH is 3·0–3·6. The impregnation may be effected in the cold, or with heat, but the condensation requires the application of heat. A light pre-tannage with Cr (<0·5% Cr_2O_3) or HCHO considerably improves the physical properties and appearance of the final leather. The resulting leathers are of good appearance, light-fastness, fulness, and suppleness; the water-resistance is poor in some cases.

The condensation of UF with tannin has also been studied—phenols are known to yield insol. resins with UF. The ternary condensation takes place, under the same conditions, equally well at ordinary and elevated temp., if sufficient time is allowed (ca. 24 hr.). Residual amounts of urea and formaldehyde are always small in relation to the initial quantities; this ensures the insolubility of the resulting derivatives. The amount of UF required varies with the tannin concerned (results are tabulated). There appears to be no relationship between the quantity of UF required and the phenolic or catecholic character of the tannin. Most tannins yield granular and fairly compact products with UF; in a few instances they are gelatinous. Treatment with sulphite does not affect the nature of the condensation product of a tannin, except in the case of mimosa, the sulphite derivative of which yields a gelatinous condensate. J.W.D.

Mould Growth on Chrome-tanned Leather, and Associated Changes in Physical Properties
R. G. Mitton and J. N. Turner
J. Soc. Leather Trades Chem., **39**, 343–358 (Nov. 1955)

Mould growth occurred freely on variously pretreated chrome leathers containing sufficient *p*-nitrophenol (I) to repress but not inhibit it, when these were stored in a saturated atmosphere; growth was less vigorous at lower R.H. The extent and nature of the moulds depended greatly on the pretreatment of the sample, and the storage conditions. One mould, not previously reported on leather, grew freely on samples containing mineral oil, and produced deep red stains—this was *Penicillium islandicum* Sopp. Some stains resulted from the reaction of I with alkali produced by moulds. Physical properties of the samples were almost unaffected by mould growth; some small changes were due to modification of the oil rather than the leather itself. J.W.D.

PATENT

Conditioning Machine for Fur Coats or the like
R. S. Schaab and J. E. Tiso *USP* 2,701,958

A combined combing and ironing machine which restores the lustre of the fur without any danger of ripping or tearing or weakening of the lining. C.O.C.

Treating Animal Skins and Similar Pervious Sheet Materials with Liquid (I p. 109)
Dyeing of Velours (VIII p. 124)
Synthetic Resinous Coatings for rendering Fabrics and Leather Water-impermeable but Vapour-permeable (X p. 126)

XIII—RUBBER; RESINS; PLASTICS

Burning Behaviour of some Cellulose Ester Film Compositions
C. J. Malm, N. G. Baumer, and G. D. Hiatt
Ind. Eng. Chem., **47**, 25121–2523 (Dec. 1955)

Additives capable of forming inorganic acids at high temperatures caused cellulose acetate film to burn more slowly and to need frequent relighting. Organic phosphate ester plasticisers varied in effectiveness as flame inhibitors according to their instability to heat and volatility. Triphenyl phosphate (b.p. 407°c.) was particularly effective and was considered to function by release of phosphoric acid at the combustion temperature. Films made unstable to heat by the presence of small amounts of phosphoric and sulphuric acids also burn less readily, but neutralisation with a strong base destroys the flame-inhibiting properties. Volatile plasticisers also counteract the flame-inhibiting action of a strong acid. W.K.R.

Water Adsorption on Polystyrene, Cellulose Triacetate, and Polyvinyl Chloride below Saturation Vapour Pressure
A. G. Day
J. Oil & Col. Chem. Assocn., **38**, 782–793 (Dec. 1955)

Rate curves have been obtained for water-vapour adsorption on polystyrene (I), polyvinyl chloride, and cellulose triacetate, in the absence of air, by direct weighing on a specially constructed microbalance. Low relative vapour pressures were used and isotherms were obtained in the range 25–50°c. The effect of specimen thickness on adsorption in I has been measured. The diffusion coefficients, activation energies of diffusion, and heats of adsorption for the three materials have been determined, and the adsorption has been considered briefly in terms of the mol. structure of the polymers. The B.E.T. multimolecular adsorption theory (Brunauer, Emmett, and Teller, *J. Amer. Chem. Soc.*, **60**, 309 (1938)) expresses the experimental isotherms satisfactorily, but evaluation of the B.E.T. energy constant does not confirm its theoretical significance. J.W.D.

Macromolecular Compounds

LXXIV—Effect of Aromatic Components on the Properties of Mixed Polyamides
V. V. Korshak and T. M. Frunze
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 551–557 (May–June 1955)

Mixed polyamides are prepared from an aromatic diamine (*o*-, *m*-, or *p*-phenylenediamine or 4-methyl-*m*-phenylenediamine), hexamethylenediamine, and a dicarboxylic acid (adipic or sebacic) and also from *m*-phenylenediamine, 6-hexanolactam, adipic acid, and sebacic acid. The dependence of the m.p. and solubilities of the polymers on composition are determined. The m.p.–composition curves are festoon-shaped (minimum m.p.).

LXXV—Dependence of the Properties of Polyamides on the Extent of Intermolecular Hydrogen Bonding
Idem
Ibid., 558–562

Mixed polyamides are prepared from ternary and quaternary mixtures of components selected from 6-hexanolactam and the hexamethylenediamine salts of succinic, azelaic, and sebacic acids. The extent of hydrogen bonding in these polymers is estimated from their m.p. as in previous investigations (*cf.* J.S.D.C., **71**, 114 (Feb.), 479 (Aug. 1955)).

LXXVI—Rôle of Exchange Reactions in Polyamide Formation
Idem
Ibid., 563–566

This is a more detailed discussion of work reported previously (see J.S.D.C., **71**, 120 (Feb. 1955)).

LXXVII—Dependence of the Properties of Aliphatic Polyamides having "Even" Repeating Units on the Structure of the Repeating Unit
Idem
Ibid., 756–761 (July–Aug. 1955)

A description is given of experimental work on which previously reported generalisations are based (see J.S.D.C., **71**, 114 (Feb. 1955)). Melting points are determined under strictly comparable conditions for the following polyamides, which are themselves prepared under comparable conditions—6,6-, 4,8-, 7-, 6,8-, 6,10-, 9-, 8,10-, 11-, and 17-nylons.

LXXVIII—Dependence of the Properties of Aliphatic Polyamides having "Odd" Repeating Units on the Structure of the Repeating Unit
Idem
Ibid., 762–765

A description is given of experimental work on which previously reported generalisations are based (see J.S.D.C., **71**, 114 (Feb. 1955)). It is shown that lengthening of the molecules of the original monomeric reactants creates more favourable conditions for the hydrogen bonding of amide groupings in polyamides.

LXXIX—Products of the Polycondensation of Dialdehydes and Diketones with Diamines and Glycols
V. V. Korshak and S. V. Vinogradova
Ibid., 925–929 (Sept.–Oct. 1955)

Linear polymers of low mol. wt. containing –CH:N·CH_2– groupings are obtained by reaction of hexamethylenediamine with terephthalaldehyde, with butane-2:3-dione, with pentane-2:4-dione, and with pyruvic acid. Reaction of terephthalaldehyde with hexane-1:6-diol gives a rubberlike polyacetal of three-dimensional structure, insol. in organic solvents. Reaction of terephthalaldehyde with ethylene glycol gives only a cyclic diacetal.

LXXX—A Case of Migrational Copolymerisation
Idem
Ibid., 930–933

Reaction of bifunctional acrylic esters (ethylene dimethacrylate, hexamethylene dimethacrylate, ethylene diacrylate) with hexamethylenediamine, with 1:3-diaminopropan-2-ol, and with ethylene glycol yields linear and three-dimensional copolymers by a migrational mechanism (transfer of a hydrogen atom in each elementary act); in these products a hydrocarbon chain is interrupted alternately by –CO·O– groupings and by –NH– or –O– linkages.

LXXXI—Mixed Polyamides containing Glutaric or Pimelic Acid Residues
V. V. Korshak and T. M. Frunze
Ibid., 934–941

A study is made of mixed polyamides derived from hexamethylenediamine glutarate or pimelate as one component and hexamethylenediamine adipate, azela-ate,

or sebacate as the other. The m.p.–composition curves are studied in the light of Korshak's views on the factors determining the extent of hydrogen bonding (see above and J.S.D.C., **71**, 114 (Feb. 1955)), various possible mutual dispositions of macromolecules within the polymer being considered. Hydrogen bonding in mixed polyamides is increased by cold drawing.

LXXXIII— Synthesis of Polyalkylene*cyclo*alkyls
V. V. Korshak and G. S. Kolesnikov
Ibid., 1090–1094 (Nov.–Dec. 1955)

Polymers prepared by the transarylation of diarylalkanes (cf. J.S.D.C., **69**, 38 (1953)), and also by the polycondensation of dihalogenoalkanes with aromatic hydrocarbons, are hydrogenated over Raney Ni with formation of polymers in which methylene or ethylene groups alternate with substituted or unsubstituted *cyclo*hex-1:4-ylene groups or with pairs of such groups. These products are much paler in colour than the original aromatic polymers.

LXXXVI— Determination of the Molecular Weights of Polyamides from the Viscosities of their Solutions in Cresol and in Methanol
V. V. Korshak and S. A. Pavlova
Ibid., 1107–1111

Viscosity measurements are made on cresol and methanol soln. of fractions obtained as already described (see J.S.D.C., **69**, 227 (1953); **70**, 477 (1954)) from a mixed polyamide, and the results are correlated with the mol. wt. of the fractions as determined by the sedimentation–diffusion method (*loc. cit.*). The relation between specific viscosity and molecular weight can be expressed by the formula—

$$[\eta] = 0{\cdot}29 \times 10^{-4}M^{1\cdot5}$$

or $[\eta] = 8{\cdot}58 \times 10^{-4}M - 0{\cdot}055$ for cresol soln.

and—

$$[\eta] = 5{\cdot}2 \times 10^{-4}M^{0\cdot9}$$

or $[\eta] = 1{\cdot}55 \times 10^{-4}M + 0{\cdot}0084$ for methanol soln.

A.E.S.

Polycondensation of Glycine Ethyl Ester
V. V. Korshak, K. T. Poroshin, and T. D. Kozarenko
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 1112–1117 (Nov.–Dec. 1955)

The results of experiments on the polycondensation of glycine ethyl ester are, in general, similar to those on DL-alanine ethyl ester (see J.S.D.C., **71**, 69 (Jan. 1955)).

A.E.S.

Reactions of Unsaturated and Macromolecular Compounds. VI— Copolymerisation of Vinylcaprolactam with Methyl Methacrylate in presence of Benzoyl Peroxide or 2:2′-Azobis[2-methylpropionitrile]
M. F. Shostakovskiĭ, F. P. Sidel'kovskaya, and A. M. Khomutov
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 919–924 (Sept.–Oct. 1955)

In presence of a free-radical initiator (benzoyl peroxide or 2:2′-azobis[2-methylpropionitrile]), mixtures of vinylcaprolactam (2-oxo-1-vinylhexamethyleneimine) (I) and methyl methacrylate (II) form copolymers in which the proportion of units derived from I is less than in the original mixture. An examination of the solubilities of the products— which are approx. intermediate between those of the hydrophilic polymer of I and of the hydrophobic polymer of II— and of the results of fractional dissolution shows that no single-component polymers are present, although the copolymers are highly non-uniform with respect to composition. A.E.S.

Polymerisation of *iso*Propyl Vinyl and *cyclo*Hexyl Vinyl Ethers in presence of 2:2′-Azobis[2-methylpropionitrile]
M. F. Shostakovskiĭ, F. P. Sidel'kovskaya, and É. S. Shapiro
Izvestiya Akad. Nauk S.S.S.R., Otdel. khim. nauk, 1085–1089 (Nov.–Dec. 1955)

Electron-microscopic Observation of Monolayers of Synthetic Linear Polymers
T. Tachibana, K. Inokuchi, and T. Inokuchi
Nature, **176**, 1117–1119 (10 Dec. 1955)

Monolayers of 6-nylon spread at 80 sq. A. per monomeric unit showed winding microfibrils. Compression to 30 sq. A. per monomeric unit produced a large number of microfibrils oriented approximately at right angles to the direction of compression. Further compression to 2 sq. A. per monomeric unit caused visible striations on the film. Material of lower molecular weight produced thicker microfibrils. Compressed films of cellulose acetate and polyvinyl alcohol showed similar behaviour, but amorphous polymers such as polyvinyl acetate and polymethyl methacrylate did not. W.R.M.

Photosensitisers for Polyester–Vinyl Polymerisation
C. M. McCloskey and J. Bond
Ind. Eng. Chem., **47**, 2125–2129 (Oct. 1955)

Compounds containing the groups—

X·C·C:C X·C·C:O X·N·C:O X·N·S(→O)$_2$

(X = halogen other than fluorine) were found to be efficient photosensitisers for initiating the polymerisation of methyl methacrylate and styrene–glycol maleate monomers by ultraviolet radiation. The naphthoyl peroxides also were active photosensitisers. W.K.R.

Photochemical Redox Polymerisation of Acrylonitrile
S. Neue
Faserforsch. und Textiltech., **6**, 450–456 (Oct. 1955)

The influence of nitrogen, oxygen, light, and temperature variations on acrylonitrile polymerisation is studied, oxalic acid being used as redox activator. Equations are given for the dependence of the induction period, initial rate of polymerisation, and molecular weight of polymer on light intensity. W.R.M.

Monomeric and Polymeric Vinyl Acetate and its Derivatives
O. Horn
Chem. and Ind., 1748–1755 (31 Dec. 1955)

The production and polymerisation of vinyl acetate are described, and uses for this polymer, polyvinyl alcohols, and polyvinyl acetals are reviewed. W.R.M.

Copolymerisation of Butyl Vinyl Ether with Vinyl Chloride
M. F. Shostakovskiĭ, V. V. Zhebrovskiĭ, and B. A. Aronov
J. Appl. Chem. U.S.S.R., **28**, 1123–1127 (Oct. 1955)

Emulsion copolymerisation of butyl vinyl ether and vinyl chloride is carried out at molar ratios of 1 : 3–5 in presence of ammonium persulphate at 30–50°c. Products containing one ether unit to 4–12 chloride units are obtained. Their solubilities in organic solvents are determined. A.E.S.

Sensitised Photopolymerisation of Styrene. I— Rapid Method of Selecting and Studying Dyes
K. Ueberreiter and G. Sorge
Z. Elektrochem., **57**, 795–800 (1953)
Chem. Abs., **49**, 16509 (28 Nov. 1955)

When styrene is photopolymerised in presence of sensitising dyes, energy transfer from an excited dye mol. occurs as well as quenching through thermal collisions. Neutral Red is three times as effective as Quinoline Yellow, Victoria Blue and Gentian Violet have average efficiency. The desensitising action of Sudan III is caused by a high amount of quenching. C.O.C.

Toluidine Red Bloom in Stoved Synthetic Finishes
G. Wormald
Offic. Dig. Federation Paint Varnish Production Clubs, **27**, 264–272 (1955)
Chem. Abs., **49**, 16456 (25 Nov. 1955)

Surface bloom on stoved synthetic enamel films containing Toluidine Red or other monoazo pigment is caused by variables in formulation or processing. The more soluble the pigment in the vehicle (bleeding) the more likely it is to bloom. Using melamine–formaldehyde resins instead of urea–formaldehyde resins reduces bloom. Bloom is related to rate of through-dry of the film. Therefore it is better to use subsurface driers rather than top driers. Type of substrate primer used is also important. A more highly pigmented and so more porous primer coat reduces tendency to bloom. Primers containing alkyds modified with soya or dehydrated castor oils are preferable to those modified with linseed or tung oil. Other factors which lower tendency to bloom are stoving at < 275°F. and application of top coat at minimum film thickness.

C.O.C.

Luminous Plastics
Z. Yoshida
J. Japan. Chem., **8**, 576–582, 661–665, 669 (1954)
Chem. Abs., **49**, 16504 (25 Nov. 1955)
Review, 37 references. C.O.C.

PATENTS

Coloured Layers of Polyvinyl Materials
FH *BP* 740,611
Plastic materials can be readily coated by forming azo dyes on them from dispersions of the dye components in aqueous polyvinyl compounds. Single-coloured or patterned effects with sharp edges can be produced. The coloured coating is very resistant to abrasion. C.O.C.

Viscosity Control of Alcohol-modified Urea Resins
Société Nobel Française *BP* 736,420
Controlled increase in viscosity with sustained compatibility with alkyds and hydrocarbon solvents is obtained in conventional alcohol-modified urea–formaldehyde condensates by refluxing with 0·4–1·5% each of pentaerythritol and maleic anhydride. E.C.

Patterned Thermoplastic Sheeting
Semtex *BP* 736,009
Patterned, e.g. marbled, effects are obtained in thermoplastic sheeting, e.g. polyvinyl chloride, by calendering a mixture of pieces of thermoplastic resin of different colours, superimposing this and consolidating the mixture on a base sheet of the same colour as one of the initial components and calendering the duplex sheet in a direction substantially at right angles to the direction of traverse of the previous operation. E.C.

Bonding of Fluorine-substituted Polyethylenes
Connecticut Hard Rubber Co. *BP* 737,286
An adhesive coating is produced on the surface of fluorine-substituted polyethylenes, e.g. polytetrafluoroethylene, by applying an aqueous dispersion of the same polymer and a solution or dispersion of an elastomer, e.g rubber, and fusing the coating, e.g. at 715°F. for 5 min. E.C.

Dispersions of Polytrifluorochloroethylene (V p. 119)
Chemistry of the Polymerisation of 6-Hexanolactam (VI p. 120)
Aromatics in Fibres and Films (VI p. 121)
Effect of Nitrogen Oxide Gases on Processed Cellulose Acetate Film (IX p. 124)
Synthetic Resinous Coatings for rendering Fabrics and Leather Water-impermeable but Vapour-permeable (X p. 126)

XIV— ANALYSIS; TESTING; APPARATUS

Determination of Minute Traces of Water by Use of Methylene Blue
F. Nesh
Anal. Chem., **27**, 1842–1843 (Nov. 1955)
The sample (1 vol.) is dissolved in CCl_4 (2 vol.), and ~ 10 mg. dry Methylene Blue (water-sol.) is added. Water is released, owing to decreased solubility in the solvent mixture (the example given is of water in hexachloroethane), and coagulates in globules in which the dye dissolves. As little as 0·03 mg. of water can be detected in this way. J.W.D.

Potentiometric Method for Karl Fischer Titrations
F. J. L. van Lamoen and H. Borsten
Anal. Chem., **27**, 1638–1639 (Oct. 1955)
Two Pt electrodes, one bright and one platinised, permit potentiometric titrations using the Karl Fischer–Johansson procedure (*Svensk Papperstidning*, **50**, 124 (1947)) with an accuracy equal to that of the dead-stop technique. A diagram of the titration cell and a photograph of the apparatus are shown. J.W.D.

Direct Determination of Moisture in Sodium Hydroxide
C. L. Dunning and C. D. Hildebrecht
Anal. Chem., **27**, 1927–1929 (Dec. 1955)
The sample is heated to ~ 350°C. in a closed Ni container, and the moisture is swept into tared tubes containing anhyd. $Mg(ClO_4)_2$ by means of a stream of dry N_2. An analysis requires ca. 45 min. There is a photograph of the apparatus. In 95% of the analyses the precision is ± 0·012%. J.W.D.

Complexometric Determination of Sulphide
P. Kivalo
Anal. Chem., **27**, 1809 (Nov. 1955)
The alkaline sulphide soln. is added to an acetate-buffered soln. containing an excess of cupric perchlorate, and the stoichiometrically pptd. CuS is filtered off, washed, and titrated in ammoniacal soln. with standard ethylenediaminetetra-acetic acid, using murexide as indicator. The method is as accurate as the classical iodometric procedure. J.W.D.

Paper-chromatographic Analysis of Mixtures of C_{12}–$_{18}$ Saturated and Unsaturated Fatty Acids
H. Wagner, L. Abisch, and K. Bernhard
Helv. Chim. Acta, **38**, 1536–1541 (Oct. 1955)

Ultraviolet Absorption of Dichromate Ions
H. Kobsa
Mh. Chem., **86**, 662–667 (Aug. 1955)

Colorimetric Submicro-method for Determination of Ammonia
P. G. Scheurer and F. Smith
Anal. Chem., **27**, 1616–1618 (Oct. 1955)
Sodium phenoxide is added to a soln. of ammonia that has been treated with HOCl, and the blue colour which develops is measured spectrophotometrically at 620 mμ. The colour density is linearly related to the ammonia concn. over the range $0–8 \times 10^{-9}$ mole/ml. The method has been used to determine the mol. wt. of cpd. whose N is directly converted to ammonia by the action of alkali. Coupled with the Kiliani cyanohydrin reaction it can be used to determine the average mol. wt. of aldehydes such as sugars and certain polysaccharides. J.W.D.

Spectrophotometric Determination of Methoxyl
A. P. Mathers and M. J. Pro
Anal. Chem., **27**, 1662–1664 (Oct. 1955)
Methoxyl is hydrolytically cleaved (with H_2SO_4) to CH_3OH, and the latter is oxidised by $KMnO_4$ to HCHO; this is condensed with chromotropic acid, and the developed colour is measured spectrophotometrically at 570 mμ. Unlike the Zeisel method, this procedure is specific for the methoxyl group. When ethanol is present, the resulting CH_3CHO interferes, and a blank determination on a soln. containing the same amount of ethanol is necessary. A micro-scale method is described. J.W.D.

Determination of Aldehydes using Unsymmetrical Dimethylhydrazine
S. Siggia and C. R. Stahl
Anal. Chem., **27**, 1975–1977 (Dec. 1955)
An excess of a soln. of unsymmetrical dimethylhydrazine in ethylene glycol or methanol (according to the class of aldehyde to be determined) is added to the sample; when reaction is complete, the excess is titrated with standard HCl in methanol. Ketones cannot be determined by this method. Aromatic aldehydes can be determined in the presence of ketones, but aliphatic aldehydes cannot. The reagent is alkaline, and is stable towards decomp. and oxidation; precision and recovery within ± 1% are possible. The method is not confined to water-soluble aldehydes. Acetals and carboxylic acids do not interfere. J.W.D.

Determination of Formic Acid in presence of Acetic Acid
B. R. Warner and L. Z. Raptis
Anal. Chem., **27**, 1783–1784 (Nov. 1955)
Formic acid is distilled azeotropically with $CHCl_3$ and then determined alkalimetrically. Separation from other organic acids, including acetic acid, is complete, and the recovery of formic acid is 95–100%. J.W.D.

Amperometric Determination of Ethylenediaminetetra-acetic Acid with Zinc Ions
W. S. Wise and N. O. Schmidt
Anal. Chem., **27**, 1469–1471 (Sept. 1955)
A soln. containing the Ca or Mg chelates of ethylenediaminetetra-acetic acid (I) is titrated amperometrically with $ZnSO_4$ at a low pH for total I; available I is determined at a higher pH to avoid displacing Ca from its chelate. If Mg is present, glycine is added to prevent the Zn displacing the Mg from its chelate. Amperometric titration curves are shown, and experimental results are cited. J.W.D.

New Spot Test for Formaldehyde
P. W. West and B. Sen
Anal. Chem., **27**, 1460–1461 (Sept. 1955)

Reagent papers are prepared by impregnating filter paper with an equilibrium mixture of potassium tetracyanonickelate and dimethylglyoxime. As little as 0·5 μg. of HCHO can be detected, and the test may be used semi-quantitatively— spots containing 0·5, 5, 25, and 50 μg. of HCHO are distinguishable from one another. Very few substances interfere, and those that do are easily suppressed; the most serious interference is from CH_3·CHO.
J.W.D.

Influence of Oxidisability of Oil-containing Substances on Rayon Materials. III— Analytical Methods
K. Täufel, R. Vogel, and A. Wicklein
Faserforsch. und Textiltech., **6**, 489–495 (Nov. 1955)

Results of the Mackey and filter-paper tests are compared. It is concluded that the filter-paper test may be successfully used to estimate the sensitivity to oxidation and ageing of oils and similar materials used in the preparation of cellulose fibres. Consideration of sensitivity to oxidation, results of the Mackey test, discoloration and washing out, and the decomposition of the cellulose during ageing leads to suggestions for the practical use of these materials.
W.R.M.

Partition Chromatography of Synthetic Detergents
F. Franks
Nature, **176**, 693–694 (8 Oct. 1955)

Synthetic detergents may be separated by ascending and circular chromatography upon filter paper impregnated with a long-chain fatty alcohol such as cetyl; the mobile phase is aq. ethanol saturated with cetyl alcohol. The chromatogram is developed by immersion in cupric acetate (0·5%) followed by spraying with a soln. of Rhodamine 6GB (0·05%). The detergent spots appear crimson on a pink background in daylight, and dark purple on a yellow background under ultraviolet radiation. R_f values are reported for Na alkyl sulphates having 12, 14, 16, and 18 C atoms in their chains, with different concn. of ethanol in the mobile phase. The method applies to Na *n*-alkyl sulphates, but with slight modifications (which are indicated) it is valid also for alkylaryl-sulphonates, *sec.*-alkyl sulphates, and quaternary ammonium and pyridinium salts; the indicator used for the latter is Bromocresol Green.
J.W.D.

Determination of the Surface Tension of Solutions of Wetting Agents
H. A. Wannow
Melliand Textilber., **36**, 1041–1042 (Oct. 1955)

Comparative measurements of surface tension are of value only if the method of determination is the same. The dimensions of the apparatus in stalagmometry must be defined.
S.R.C.

Investigations on the Standardisation of Test Methods for Textile Auxiliaries. VIII— Determination by the "Emulsifying Method" of the Stabilities to Hard Water, Acids, and Metallic Salts of Surface-active Compounds
F. Nölke
Melliand Textilber., **36**, 1073–1074 (Oct. 1955)

The method is based on the influence of Ca^{2+}, Mg^{2+}, H^+, and Fe^{3+} on the emulsifying power of the surface-active body with respect to CCl_4.
S.R.C.

Standardisation of Testing Methods for Textile Auxiliaries. IX— Assessment of Enzymatic Desizing Agents for Goods sized with Starch
W. Jülicher
Melliand Textilber., **36**, 1293–1294 (Dec. 1955)

Details are given of tests carried out under the optimum conditions of temperature and pH value for bacterial, pancreatic, and malt amylases, using a standard iodine-staining technique on the desized patterns and comparing the results with a standard violet scale prepared in a standard manner. The transience of the colour obtained by iodine and staining can be overcome by photographing the stained pattern alongside the violet scale.
S.R.C.

Volumetric Determination of Soluble Silicates in Detergents
A. M. Lawson, L. A. Jones, and O.T. Aepli
Anal. Chem., **27**, 1810–1811 (Nov. 1955)

The method is based on the reactions—

$$Na_2SiO_3.H_2O + 2HCl \rightarrow 2NaCl + H_2SiO_3 + H_2O$$

$$H_2SiO_3 + 6NaF + H_2O \rightarrow Na_2SiF_6 + 4NaOH$$

$$NaOH + HCl \rightarrow NaCl + H_2O$$

The direct titration of the liberated NaOH with acid is very slow, and the end-point obscure. The addition of an excess of standard acid and ethyl alcohol increases the reaction rate, so that no waiting period is necessary. The use of Xylene Cyanol FF–Methyl Red indicator increases the accuracy of the end-point, so that the method can be used by non-technical personnel. Carbonates, phosphates, and wetting agents do not interfere. The results obtained agree with those obtained by gravimetry within ± 0·05% SiO_2.
J.W.D.

Determination of Trace Metals in Oils
J. T. Horeczy, B. N. Hill, A. E. Walters, H. G. Schutze, and W. H. Bonner
Anal. Chem., **27**, 1899–1903 (Dec. 1955)

The metallic derivatives of porphyrins are volatile at high temp., and this leads to serious inaccuracy in their determination by dry-ashing procedures. A wet-ashing method is given, whereby this loss is eliminated and good accuracy ensured. Information on the preparation of tetraphenylporphyrin and its metallic derivatives is given in full detail.
J.W.D.

Titration of Papain
P. Mesnard and R. Babin
Bull. Soc. pharm. Bordeaux, **91**, 214–218 (1953)
Chem. Abs., **49**, 12772 (25 Sept. 1955)

Determination of papain by making an extract and determining the minimum rotating power is not precise and is inconvenient. A better way is to determine the laevorotation of fibrin hydrolysed by papain, the strength of the enzyme being calculated from S/E = wt. of substrate taken/wt. of papain.
C.O.C.

Separation and Identification of Phenols by Paper Chromatography
J. Green and S. Marcinkiewicz
Nature, **176**, 1172–1173 (17 Dec. 1955)

A two-dimension technique in which the first-dimensional separation is obtained on $ZnCO_3$-impregnated paper, using benzene as the developing solvent; the second-dimensional separation takes place on the same paper, using ethyl oleate as the stationary phase, and aq. ethanol as the developing solvent. Both separations are obtained by the ascending method. Many closely related phenols are separated by this method. The R and R_f values of 16 phenols are reported.
J.W.D.

X-Ray Diffraction Patterns of Phenols
L. J. E. Hofer and W. C. Peebles
Anal. Chem., **27**, 1852–1856 (Dec. 1955)

Crystalline phenols may be positively identified by their X-ray diffraction patterns. If the m.p. of the phenol is reasonably high, it is not necessary to produce the phenyl *iso*cyanate derivative in order to obtain a diffraction pattern. It is also unnecessary to crystallise the specimen until the literature m.p. is attained. The diffraction patterns of isomers and closely related cpd. are distinctly different. The procedure is detailed, and there are copious data relating to 51 phenols.
J.W.D.

Rapid Detection of Aniline Vapours in Air
W. A. Riehl and K. F. Hager
Anal. Chem., **27**, 1768–1769 (Nov. 1955)

Test-paper strips impregnated with the liquid or vapour of a furfural–acetic acid mixture (4% by vol. in acetic acid) turn from white to pink or red in the presence of aniline vapour; the method may be used semi-quantitatively within the range 5–150 p.p.m. Aniline vapour is toxic in air, in concn. > 5 p.p.m. by vol. The method is virtually specific.
J.W.D.

Chromatographic Separation and Quantitative Estimation of 131-Iodine-labelled Derivatives of Sterols, Amines, Acids, and Aldehydes
W. M. Stokes, W. A. Fish, and F. C. Hickey
Anal. Chem., **27**, 1895–1898 (Dec. 1955)

Mixtures of cpd. labelled with ^{131}I have been chromatographed on a column of silicic acid (two-thirds) and Celite 503 (one-third). The course of the development is followed at all stages, by automatically scanning the column and recording the distribution of the associated γ-activity. The apparatus is illustrated. The components of selected pairs of derivatives of sterols, amines, acids, and aldehydes have been successfully separated and estimated. J.W.D.

Displacement of the Nitro Group during Determination of Nitrophenols and Nitroanilines by the Koppeschaar Method
L. D. Johnson, W. M. McNabb, and E. C. Wagner
Anal. Chem., **27**, 1494–1498 (Sept. 1955)

Picric acid is converted by Br to 6-bromo-2:4-dinitrophenol, even under the mildly alkaline conditions of the Koppeschaar method (*Z. anal. Chem.*, **15**, 233 (1876)). Such displacements of nitro groups, in picric acid and other cpd., may be effected by free Br or by HOBr; free HNO_3 is produced in either instance. A full study of this class of reaction is reported, and mechanisms are proposed. A procedure is described whereby mixtures of 2:4-dinitrophenol and picric acid may be analysed; both phenols are initially titrated acidimetrically, the Koppeschaar method is applied to the titrated liquid, and the picric acid is calc. by difference. J.W.D.

Rapid Colorimetric Determination of Benzidine
R. G. Rice and E. J. Kohn
Anal. Chem., **27**, 1630–1631 (Oct. 1955)

Benzidine is oxidised with $KMnO_4$ in presence of HNO_3, and the developed colour is measured spectrophotometrically using a 410–490 mμ. filter. The amount of $KMnO_4$ soln. required is critical, but small amounts of HCl have no effect on reproducibility. The average precision of the method is $\pm$ 0·13 g./litre, and the average deviation between the method and a gravimetric procedure is $\pm$ 0·19 g./litre for soln. containing 10–25 g. of benzidine dihydrochloride per litre. J.W.D.

Evaluation of Azo Dyes by the Knecht–Hibbert Method
J. Cegarra Sánchez and J. Ribé Pons
XXVIII Congrès International de Chimie Industrielle
Chim. et Ind., **74**, 180 (Oct. 1955)

The Knecht–Hibbert method has been criticised because of divergent results in the evaluation of direct dyes. The present authors have examined statistically results obtained on azo dyes and the influence of each condition on the results. The limits of application of the method and the degree of precision for a series of azo dyes are discussed. S.R.C.

4′-Nitroazobenzene-4-carboxyl Chloride as Reagent for Alcohols
E. Hecker
Chem. Ber., **88**, 1666–1675 (Nov. 1955)

This substance is readily prepared from ethyl *p*-nitrosobenzoic acid and *p*-nitroaniline. Its esters are intensely coloured and easily hydrolysed, and they have a higher m.p. and greater crystallising power than the isomers with the nitro group in position 2′ or 3′. H.E.N.

Measurement of Fluorescent Spectra
S. K. K. Jatkar and B. N. Mattoo
J. Univ. Poona, Sci. & Technol., (6), 67–73 (1954)
Chem. Abs., **49**, 15498 (25 Nov. 1955)

A fluorometer for measuring fluorescent spectra consists of a continuous ultraviolet source, direct top illumination of the fluorescent solution, a Fuess constant-deviation spectroscope as dispersing element and an IP28 photomultiplier tube with amplifier as detector. The sensitivity of the apparatus is of the order of 10^{-6} g./l. of quinine sulphate in 0·1 N-H_2SO_4. Resolution, checked with anthracene in alcohol, is satisfactory. C.O.C.

Chromatography of Dye Intermediates
III—Identification and Separation of Anthraquinonesulphonic Acids by Paper Chromatography
M. Večeřa, J. Gasparič, and J. Borecký
Chem. Listy, **49**, 706–708 (1955)
Chem. Abs., **49**, 12199 (25 Sept. 1955)

Isomeric anthraquinone-sulphonic and -disulphonic acids can be identified, the purity of technical samples tested, and the course of the sulphonation followed by using Whatman No. 4 paper with butyl alcohol–ammonia–water (2 : 1 : 1— I) or butyl alcohol–pyridine–water (3 : 1 : 1— II) as solvents. Detection is by fluorescence in ultraviolet radiation. R_f values in I and II at 21°c. for the following sulphonic acids are: 1— 0·65, 0·57; 2— 0·78, 0·72; 1:5— 0·04, 0·07; 1:6— 0·09, 0·15; 1:7— 0·14, 0·19; 1:8— 0·26, 0·35; 2:6— 0·19, 0·29; 2:7— 0·19, 0·29. C.O.C.

IV—Paper Chromatography of Dinitrobenzenes, Dinitronaphthalenes, and Dinitroanthraquinones
J. Franc
Chem. Listy, **49**, 872–875 (1955)
Chem. Abs., **49**, 12836 (25 Sept. 1955)

Dinitro derivatives of benzene, naphthalene, and anthraquinone were separated by using Whatman No. 4 paper soaked with petroleum and a 20 : 14 : 1 mixture of ethyl alcohol–water–acetic acid as the mobile phase. Detection was with fluorescein or Ehrlich reagent under ultraviolet radiation. The R_f values, except for *o*-dinitrobenzene, coincided with those calculated from the equation $R_f = K + 0{\cdot}1\mu$. With *o*-dinitrobenzene $R_f = 0{\cdot}76$, *m*-dinitrobenzene 0·56, 1-nitronaphthalene 0·33, 1:5-dinitronaphthalene 0·00, 1:8-dinitronaphthalene 0·80, 1:6-dinitroanthraquinone 0·43, 1-nitroanthraquinone 0·17, 1:5-dinitroanthraquinone 0·00, 1:8-dinitroanthraquinone 0·77, 1:7-dinitroanthraquinone 0·70, and anthraquinone 0·28. C.O.C.

Anthraquinone and Anthrone Series. XVI—Absorption Spectra of the Leuco Derivatives of Anthraquinone, 1- and 2-Aminoanthraquinones, and certain Acylaminoanthraquinones in Alkaline Solutions
M. R. Padhye, N. R. Rao, and K. Venkataraman
J. Sci. Ind. Research (India), **13 B**, 759–763 (1954)

Identification of Dyes in Boot Polishes and Shoe Creams
J. Deshusses and P. Desbaumes
Mitt. Lebensm. Hyg., **46**, 258–260 (1955)

In a paper-chromatographic method the water-soluble dyes are separated by using two ammoniacal solvents and the water-insoluble dyes by using a mixture of 80 parts of 85% formic acid and 20 parts of water. C.O.C.

Detection of Artificial Food Dyes in Confectionery
J. Eisenbrand
Zucker- u. Süsswarenwirtsch., **8**, 551–557 (1955)
Chem. Abs., **49**, 12737 (25 Sept. 1955)

A series of colour tests for detecting the 18 water-soluble food dyes commonly used in German confectionery. C.O.C.

Oxidative Degradation of Carotenoids
N. F. Holyer and B. C. L. Weedon
Chem. and Ind., 1219 (24 Sept. 1955)

Degredations may be carried out on the 10–100 mg. scale by the procedure of Karrer *et al.* (*Helv. chim. Acta*. **13**, 1084 (1930)), and the resulting diacids readily detected by paper chromatography of as little as one-tenth of the products; the acids are identified by carrying out mixed chromatograms with authentic specimens. The method is fully described. J.W.D.

Chemical Analysis of Zinc Yellow
D. Barber Cubas, A. Cheriguian Alvarez, and L. Yzu Muñoz
XXVIII Congrès International de Chimie Industrielle
Chim. et Ind., **74**, 173 (Oct. 1955)

A critique of A.S.T.M. D-445–51 and D.T.D. 377A, followed by a description of a method based on precipitation with mercurous nitrate, which saves time and reagents and obviates the need for using sulphuric acid. S.R.C.

Rapid Determination of the Dry Covering Power of Paints and Pigments by the Contrast Relations Method
J. M. Múgica, F. Escribano, and D. Lozano
XXVIII Congrès International de Chimie Industrielle
Chim. et Ind., **74**, 174 (Oct. 1955)

A modification to the "Method of Measuring Dry Hiding Power of Paints" (29th Annual Meeting of the Federation of Paint and Varnish Production Clubs in Atlantic City, Nov. 1951), revised by Subcommittee 40 of the New York Paint and Varnish Production Clubs Technical Committee, is claimed to make considerable savings in time and material. S.R.C.

Direct Volumetric Determination of Total Zinc in Mixed Paint Pigments with Ethylenediaminetetra-acetic Acid
M. H. Swann and M. L. Adams
Anal. Chem., **27**, 2005 (Dec. 1955)

The pigment is treated with a buffered soln. of NH_4Cl and NH_4OH, and insol. components are filtered off; the soln. is titrated with EDTA, using Eriochrome Black T as indicator. Few of the metals that can be determined in this way are found in mixed paint pigments, and of these only zinc (except as ZnS) passes into soln. A few Cr pigments dissolve also, but these do not interfere. When ZnS is present, it can easily be detected by its reaction with acid, and a modified procedure is described in which it may be included in the determination. J.W.D.

Examination of Textiles in Ultraviolet Radiation
N. Kuhn
Z. ges. Textilind., **57**, 1234–1236 (5 Oct. 1955)

Differences in fluorescence displayed in ultraviolet radiation are of value when examining samples of cotton for degree of ripening, presence of impurities, and damage, and also for differentiation of various types of textile fibres and impurities present, especially mineral oil. B.K.

Determination of Carboxyl Groups in Cellulose
O. Samuelson and A. Wennerblom
Svensk Papperstidning, **58**, 713–716 (15 Oct. 1955)

A modification of the method of Neale and Stringfellow (*Trans. Faraday Soc.*, **33**, 85 (1937)). The cellulose is filtered off at pH ~ 8, and the filtrate is titrated to a lower, pH to correct for the CO_2. The initial adjustment is made to the phenolphthalein end-point, and in the second titration a mixed indicator (methyl red–bromocresol green) is used. J.W.D.

Iodine Value of Cellulose
V. I. Sharkov and O. A. Dobush
J. Appl. Chem. U.S.S.R., **28**, 994–999 (Sept. 1955)

The experimental work described shows that the use of the I.V. by some workers as a measure of the content of terminal aldehyde groups in celluloses and hydrocelluloses is not justified, the results being, in general, much too low. The I.V. obtained depends greatly on the state of swelling of the cellulose, and, after oxidation in the course of the I.V. determination, cellulose can be oxidised further in a second determination. A.E.S.

Use of Oil Immersion for the Analysis of Streaks in Fabrics due to Differences in Pigmentation of Fibres
N. Iwanow and R. Schneider
Bull. Inst. Text. France, (56), 39–43 (Oct. 1955)

Streaks in fabrics due to differences in pigmentation of fibres are made more clearly visible by immersion of the samples in liquid paraffin. The method is applied— (*a*) to streaks in blends of spun viscose staple rayons of different degrees of milkiness; (*b*) to streaks in blends of spun rayons of different degrees of delustring. 12 photographs. J.C.F.

Orientation of Fibres in Microscopical Preparations to facilitate Statistical Measurements in Polarised Light
N. Iwanow and R. Schneider
Bull. Inst. Text. France, (56), 45–50 (Oct. 1955)

A modification of the method of Larose (*J. Textile Inst.*, **42**, P 104 (1951)) for obtaining preparations of parallel fibres by application of a high-voltage field. Short lengths of cellulosic fibres cut in a microtome are impregnated with sodium chloride, dried, and mounted in amyl acetate containing a small amount of cellulose nitrate. An alternating field of 6 kv. is applied until the solvent evaporates. The fibres are oriented end-to-end in chains. Applications are described to (*a*) the determination of the degree of maturity of cottons, and (*b*) the characterisation of viscose rayons by the differential dyeing test. Six photomicrographs. J.C.F.

Measurement of the Extent of Delustring of Filament Fabrics. II— Reflection of Unpolarised Light
R. Jeffries
J. Textile Inst., **46**, T 759–T 777 (Dec. 1955)

Reflection characteristics of fabrics woven from low-twist filament yarns are studied, and it is shown that the main features of the reflection can be explained in terms of the reflecting properties of the warp and weft considered separately as pads of yarn. In measuring the effect of delustring the previously described method (idem, *ibid.*, **46**, T 391 (1955)), of rotating an illuminated sample in its own plane and obtaining an orientation–intensity curve at a selected angle of collection, is proved. The unscattered warp reflection, the unscattered weft reflection, and the scattered reflection can be separately identified on the curve, and the ratio of unscattered to scattered light is used as an index of lustre. J.W.B.

Determination of Lignin in Flax
J. F. Couchman
J. Textile Inst., **46**, T 735–T 741 (Dec. 1955)

It is shown that determination of lignin in both wax-free and alkali-extracted flax is satisfactorily carried out by digesting 1 g. material for 2 hr. at 20°C. with 20 ml. H_2SO_4 of concn. 71·8–72·0% (by wt.). Care must be taken to disperse the fibre in the acid; only a small amount of lignin is dissolved. The recovered lignin is separable into two fractions by treatment with acid sodium chlorite (0·7%) and in this respect differs from jute lignin. The fraction soluble in sodium chlorite contains nearly all the methoxyl groups, and it is suggested that it is mainly of the "middle lamella" lignin, that in the cell wall being insoluble. J.W.B.

New Extraction Apparatus for Determination of Wool Grease Content
L. Mauri
XXVIII Congrès International de Chimie Industrielle
Chim. et Ind., **74**, 169–170 (Oct. 1955)

The time required for a normal Soxhlet extraction is reduced to one-fifth by employment of extraction apparatus incorporating refrigeration. S.R.C.

Electrical Resistance of Synthetic and Cellulose Acetate Fibres
G. E. Cusick and J. W. S. Hearle
J. Textile Inst., **46**, T 699–T 711 (Nov. 1955)

A technique is described for measuring resistances along the length of fibres, up to 10^{15} ohms, and results for cellulose acetate and a large number of synthetic fibres, determined over a range of R.H., are presented. Comparison of different fibres is difficult because there appear to be variations in resistance between different samples of the same fibre, according to its history. Moreover, the resistance of the most hydrophobic fibres cannot be measured below about 75% R.H. J.W.B.

Qualitative Analysis of Synthetic Fibres
S. Schwenkedel
Textil Praxis, **10**, 918–920 (Sept. 1955)

A series of tests for the identification of synthetic fibres include treatment with organic solvents, staining reactions, dry distillation, and burning and microscopical tests. B.K.

Wearing and Washing Trials with Garments and Household Linens made from Synthetic Fibres
O. Viertel
Textil Praxis, **10**, 1042–1046 (Oct. 1955)

Practical washing trials are described to illustrate some of the problems involved in removal of soiling from shirts, underwear, and household linens made from Perlon and other synthetic fibres. B.K.

Laboratory-scale Tumbler-type Washing Machine
D. G. Stevenson
J. Textile Inst., **46**, T 677–T 683 (Nov. 1955)

It is considered that the machines available for laboratory-scale studies of detergency are far from ideal,

in that they do not reproduce commercial laundering conditions at all closely; e.g. they usually operate at much higher liquor : material ratios. A brief mathematical survey is presented of what happens to material in a commercial machine, i.e. velocity of impact against the drum walls, etc., and this is used to design a laboratory machine which, by virtue of predetermined higher speeds, will reproduce such forces. It comprises two wash drums, 1½ in. × 4 in. in diameter, mounted vertically on a rotor arm at 4 in. radius, and rotated by sprocket chain drive from bevel gears meshing with a stationary gear on the central pillar. Thus the drums rotate in a vertical plane while being whirled round in a horizontal plane. They are fitted internally with ½-in. lifters on the periphery at 120° separation, and the entire assembly fits into a large drum the walls of which contain heater elements, permitting temperature control within about 3°C. The machine takes 17 g. fabric to 120 ml. liquor, and trials are described on soiled cotton twill, using urano-uranic oxide pigment, soiling being assessed by means of a β-particle counter.

J.W.B.

Measuring the Whitening Effects of Fluorescent Brightening Agents and Perborate Bleaches on Cotton
M. S. Furry and P. L. Bensing

Amer. Dyestuff Rep., **44**, P 786–P 790 (7 Nov. 1955)

Fluorescent and non-fluorescent whitening effects in fabrics are measured by means of two different reflectometers modified either to include or to exclude ultraviolet radiation from the light source. Undyed cotton fabrics are laundered repeatedly with different detergent solutions containing fluorescent brightening agents and perborate bleaches. Whitening effects as evaluated by the two instruments are comparable and conform well with visual observations. Sodium perborate greatly reduces greyness and yellowness in unbleached fabric, whereas the two fluorescent agents used with the detergents reduce yellowness and greenness but have little effect on greyness. Perborate also enhances the fluorescent effect, in some cases towards the red. With bleached fabric perborate produces less change than fluorescent agents, though by using a higher amount effective whitening results plus, in some instances, fluorescence, suggesting that chemical modification of the fibre may have occurred. Soap and fluorescent agent give greater whiteness than detergent and agent, the latter tending to redness and blueness.

J.W.B.

Use of Fade-Ometer for Light Fastness Assessment
H. Jörder and G. Müller

Textil Praxis, **10**, 920–923 (Sept. 1955)

A discussion on the conditions influencing the efficiency of the Fade-Ometer including temp., humidity, and air filtration.

B.K.

Factors influencing Quantitative Determination of Methylpentoses and Ketohexoses with Anthrone
J. R. Helbert and K. D. Brown

Anal. Chem., **27**, 1791–1796 (Nov. 1955)

The optimum temp. for developing the anthrone colour of any carbohydrate is not 100°C., as is generally supposed; for methylpentoses (I) and ketohexoses (II) it is 70°C. and 60°C. respectively. Optimum heating temp. appears to be more characteristic of a given type of monosaccharide than the optimum heating time. The time factor is particularly important in the case of I and II, the developed colours being less stable than those of aldohexoses. By controlling time and temp. within indicated tolerances, I and II can be estimated with a precision comparable to that reported for glucose.

J.W.D.

Rapid Estimation of Dialdehyde Content of Periodate Oxystarch through Quantitative Alkali Consumption
B. T. Hofreiter, B. H. Alexander, and L. A. Wolff

Anal. Chem., **27**, 1930–1931 (Dec. 1955)

Under standardised conditions, one mole of alkali (NaOH) is consumed per mole of dialdehyde in the oxystarch. The reaction conditions are critical. There is good agreement between results obtained by this method and those by other procedures over a wide range of dialdehyde contents. A single determination requires only 10 min.

J.W.D.

Fading of Photographic Colour Images
Y. Tomoda and N. Nakamura

Repts. Govt. Chem. Ind. Research Inst. Tokyo, **50**, 63–69 (1955)

Chem. Abs., **49**, 15577 (25 Nov. 1955)

When tested for 13 hr. in a Fade-Ometer, standard cinematograph colour films exposed for 10 sec. to sunlight and developed with a colour developer containing a chromogenic compound showed that the yellow developed from $C_6H_5NHCOCH_2COCH_3$ was the most unstable and that from $C_6H_4OHCH_2COOC_2H_5$ slightly less unstable. The magenta from 1-phenyl- or 1-(*p*-nitrophenyl)-3-methyl-5-pyrazolone was faster than that from *p*-$NO_2C_6H_4CH_2CN$ or 3-Br-4:5- or 6-$NO_2C_6H_3CH_2CN$. The blue from 2:4-dibromo-1-naphthol was the fastest of all and that from 1-naphthol, thymol or 8-quinolinol fairly fast. Commercial three-layer colour films similarly tested gave colours which were generally faster than the above; in these cases the yellow was the least fast, the magenta fairly fast and the blue the fastest. Non-diffusing (long aliphatic chain) chromogenic compounds do not necessarily develop fast colours.

C.O.C.

Urea- and Melamine-Formaldehyde Compounds with special reference to Etherified Monomers (Precondensates)
W. Bitterli

Textil-Rund., **10**, 653–658 (Dec. 1955)

A summary of qualitative tests for urea- or melamine-formaldehyde monomers and cured resins on finished fabrics is given. A modified Zeisel procedure is described which permits the detection of alkoxyl groups in etherified derivatives after acid hydrolysis and removal of interfering formaldehyde by oxidation (Ag_2O) or dimedone. For monomers which are not etherified, a quantitative formaldehyde determination allows the mol. ratio of formaldehyde to urea or melamine component to be calculated or read from a curve which is given.

J.C.F.

Amperometric Titration of Viscose
M. Wronski

J. Polymer Sci., **19**, 213–214 (Jan. 1956)

Amperometric titration, using the dropping mercury electrode, is more precise and rapid than potentiometric titration. Details of typical titrations are given.

W.R.M.

Cuprammonium Viscosity of Cellulose
Z. L. Zosim

Tekstil. prom., **15**, 34–36 (March 1955)

Higher viscosity figures are obtained when cotton (grey or bleached) is treated with a solution of a wetting agent before dissolution in cuprammonium soln. This effect is ascribed to the displacement of air usually retained by the cotton. Still higher viscosities are obtained when air is removed from cotton *in vacuo* before the dissolution process.

A.E.S.

Influence of Oxygen on the Cuprammonium Viscosity of Cellulose
A. A. Morozov and Z. L. Zosim

Ukrain. khim. zhur., **19**, 687–696 (1953)

Chem. Abs., **49**, 16427 (25 Nov. 1955)

Cotton cellulose— untreated (I), extracted with equal vol. of ethanol and benzene (II), and bleached (III)— is dissolved in cuprammonium soln. (1·0% and 0·1%) in presence of air at pressures ranging from atmospheric to $2·5 \times 10^{-3}$ mm. The degradative effect of oxygen is greater in 1·0% soln. than in 0·1% soln. Extrapolation of mol. wt. to zero oxygen content gives $1·2–1·3 \times 10^6$ for I, $1·4–1·5 \times 10^6$ for II, and $0·55–0·60 \times 10^6$ for III. A 0·1% cuprammonium soln. is recommended for use in viscosity determinations on cellulose. (See also preceding abstract.)

A.E.S.

Rapid Method for the Determination of α-Cellulose
E. I. Pozhitkova and I. A. Nagrodskii

Bumazh. prom., **30**, 12–13 (April 1955)

Chem. Abs., **49**, 15229 (10 Nov. 1955)

Air-dried pulp (~ 0·3 g.) is mercerised in 10 ml. of 17·5% NaOH at 20°C. for 30 min. and washed with 9·5% NaOH. The α-cellulose is separated and is estimated by oxidation with standard dichromate in presence of H_2SO_4 and back titration with ferrous salt. The hemicelluloses are similarly determined in the filtrate and washings. The time required for the analysis is 1 hr., and duplicate tests agree within 0·6% of α-cellulose.

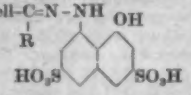
A.E.S.

Volumetric Determination of Hemicellulose with Potassium Dichromate
Y. Tomari *J. Soc. Textile Cellulose Ind. Japan*, **11**, 727–732 (Nov. 1955)

Different methods have been proposed for the volumetric determination of hemicellulose with potassium dichromate, and the results obtained from them are not completely coincident. The optimum conditions, which produce the highest value of hemicellulose, are studied. It is confirmed that the method not only yields a nearly theoretical value, but also is universally applicable, for its conditions are common regardless of the concentration of hemicellulose. Therefore the method is suitable for factory control. At the same time, the effects of each factor on the results are investigated, and the reason for the variability of previous methods is discussed. AUTHOR

Colour Reaction for Aldehyde and Ketonic Groups in Oxycelluloses
J. Pinte and P. Rochas
Bull Inst. Text. France, (57), 43–52 (Dec. 1955)

The reaction due to Müller (*Helv. Chim. Acta*, **22**, 217 (1939); *Melliand Textilber.*, **26**, 93 (1945)) detects only aldehyde groups in oxycelluloses. In the present modification, the hydrazine analogue of H acid is used to form the hydrazone derivative of the carbonyl group—



Diazotised H acid can then be coupled *ortho* to the OH group to give a coloured derivative. The reaction is applicable to both aldehyde and ketonic carbonyl groups, and is thus more useful for assessing degradation which involves the formation of such groups. The application of the reaction to various modified rayons is described. J.C.F.

Detection of Amino Acids on Paper Chromatograms
J. A. Cifonelli and F. Smith
Anal. Chem., **27**, 1501–1502 (Sept. 1955)

The amino acids are first oxidised by periodate, and then detected on paper chromatograms; four procedures are given for this, using two different benzidine reagents, and a periodate–NaI reagent in both neutral and alkaline conditions, these latter being used in conjunction with starch–iodide. Results obtained by these four methods with 15 amino acids are tabulated, and R_f values are listed for 18 amino acids with *tert.*-amyl alcohol–1-propanol–water (the irrigating solvent) in two different ratios. The two benzidine reagents may be successively applied to the same chromatogram. J.W.D.

Quantitative Determination of Amino Acids by Circular Paper Chromatography
K. Krishnamurthy and M. Swaminathan
Anal. Chem., **27**, 1396–1399 (Sept. 1955)

Chromatograms of the unknown amino acid mixture and a known mixture of standard amino acids are developed side by side on the same paper. The solvent mixtures used are phenol–1-butanol–acetic acid saturated with water, benzyl alcohol–*tert.*-amyl alcohol saturated with water, *m*-cresol saturated with pH 8·4 buffer, 1-butanol–acetic acid saturated with water, and phenol saturated with pH 12 buffer. For quant. determinations, the chromatograms are treated with ninhydrin, and the coloured bands due to each amino acid in the standard and unknown chromatograms are cut. The colour is extracted with 75% ethanol, and estimated spectrophotometrically at 560 mμ. There is a sketch of the apparatus, and five photographs of actual chromatograms are shown. The method yields highly reproducible results in the determination of the amino acids of casein, and these agree with those reported by other workers. The advantages claimed for the method are speed, clarity, and compactness of the amino acid zones, and the use of cheap and commonly available solvents. J.W.D.

Colorimetric Method for the Estimation of Tyrosine
E.-R. Fritze and H. Zahn
Melliand Textilber. **36**, 1136–1139 (Nov. 1955)

The procedure is given in detail for a test based on the reaction of tyrosine with mercuric sulphate in sulphuric acid and photoelectric comparison against a standard pure tyrosine (L Tyrosine, Merck). The validity and limiting concentrations of the Lambert–Beer law are tested, and the experimental error is examined statistically. The test is designed to cover a wide range of tyrosine concentrations, and examples of its use are quoted. S.R.C.

Infrared Spectra of 3-Phenyl-2-thiohydantoins of Amino Acids and their Application to Identification of *N*-Terminal Groups in Peptides
L. K. Ramachandran, A. Epp, and W. B. McConnell
Anal. Chem., **27**, 1734–1737 (Nov. 1955)

Infrared spectra of the phenylthiohydantoins of 22 amino acids are recorded. Five peptides (DL-alanylglycylglycine, L-leucylglycine, DL-phenylalanylglycine, DL-histidylhistidine, and γ-glutamylcysteinylglycine) have been degraded by the Edman method; the value of infrared spectra for the positive identification of *N*-terminal amino acid residues is demonstrated. J.W.D.

Polarographic Determination of Phthalic Anhydride in Alkyd Resins
P. D. Garn and E. W. Halline
Anal. Chem., **27**, 1563–1565 (Oct. 1955)

Measurement of the Molecular Weight of Polymers by the Light-scattering Method— Experimental Procedure and Interpretation of Results
D. Cleverdon, L. Harvey, D. Laker, and P. G. Smith
J. Appl. Chem., **5**, 503–509 (Sept. 1955)

Determination of Water in Polyamide Chips by the Karl Fischer Method
H. Sieber
Faserforsch. und Textiltech., **6**, 421–422 (Sept. 1955)
H. Reimschüssel
Ibid., **6**, 568 (Dec. 1955)

PATENTS

Testing the Adhesive Properties of Sizes
Gebrüder Sucker *BP* 740,922

The condition of a sizing liquid is determined by wetting two surfaces of a testing instrument with it, pressing the two surfaces together and measuring the force required to pull them apart. C.O.C.

Measuring the Tackiness of a Sheet
Dunlop Rubber Co. *BP* 741,214

A device especially suitable for use with rubber or rubberised fabric. C.O.C.

Measuring the Wrinkle Resistance of Fabrics
Celanese Corpn. of America *USP* 2,701,962

A sample of fabric is enclosed within a flexible member which is periodically collapsed on to the fabric and moved away from the fabric so as to wrinkle it. After a definite number of cycles the fabric is examined either visually or in any other suitable manner to determine its resistance to wrinkling. C.O.C.

Fabric Analyser
G. A. W. Abbott *USP* 2,701,980

The fabric is photographed in enlarged silhouette preferably against a calibrated grid. The photograph shows the fabric in detail so that structural characteristics and defects can easily be observed. It also serves as a permanent record of the analysis. C.O.C.

Wear-testing of Textiles
Linen Industry Research Assocn. *BP* 743,300

Modification of *BP* 686,330 (J.S.D.C., **69**, 180 (1953)). A helix of hardened wire placed in a groove formed eccentrically in the surface of a rotatable base plate and projecting above this surface is used as the abradant. C.O.C.

Spray Gun (I p. 109)

Effect of Micellar Size on Physicochemical Properties of Surface-active Agents (III p. 109)

Preparation of an *N*-Hydroxymethyl Derivative of Steelon (6-Nylon) soluble in Methanol and Ethanol (III p. 110)

Absorption Spectra of Dyes in Solution (IV p. 112)

Technical Developments in Pigments and Paints during 1955 (V p. 118)

End-group Titration in the Determination of the Heterogeneity of Perlon L (VI p. 121)

Dyeing Vinylon black with Oxidation Dyes. IV— Effect of Combined Oxidation and the Damage to the Fibres (VIII p. 123)
Effect of Nitrogen Oxide Gases on Processed Cellulose Acetate Film (IX p. 124)
Interpretation and Measurement of Contact Angles of Water on Paper (XI p. 126)

XV— MISCELLANEOUS

Correction of Anomalous Colour Vision by the aid of Colour Filters

G. N. Rautian

Doklady Akad. Nauk S.S.S.R., **104**, 219–222 (11 Sept. 1955)

Yustova (*ibid.*, **81**, 1051 (1951)) showed that the vision of some anomalous trichromats could be corrected by the use of colour filters. The results are given of attempts to correct the vision of deuteranomalous and protanomalous observers by Yustova's method. For many observers, including five out of six protanomalous trichromats, full correction could not be effected. A.E.S.

Distribution of Colour-sensitive Receptors in the Human Fovea

M. S. Smirnov

Doklady Akad. Nauk S.S.S.R., **103**, 427–429 (21 July 1955)

Supporters of the view that each cone of the fovea carries a single receptor sensitive to one primary spectrum range only have explained the experimentally observed independence of visual acuity on colour by the occurrence of clusters of cones of given spectral sensitivity [see White and Vickerstaff, J.S.D.C., **61**, 213 (1945)]. The author gives a brief description of some of his experiments, the results of which show that such clusters are absent. The results can be readily explained only on the assumption that every foveal cone carries red-, green-, and blue-sensitive receptors. Experiments on visual acuity confirm that for vision by red light acuity is such that the red-sensitive receptors cannot be more than 2·6–2·7 μ. apart, in close accord with the experimentally determined spacing of foveal cones (2·5–3·0 μ.). Results for green light are substantially the same. Aberrations of various kinds make accurate determinations in blue light very difficult, but it is established that a blue-sensitive receptor must be present in most of the foveal cones (this proves that the prevailing view of the existence of "blue-blindness" in the fovea is incorrect). In one series of experiments, the eye is "fixed" on a small source of red or blue light (angle subtended at eye $\sim$ 0·3′), so that the image moves over a small area of the retina. The light is interrupted for short periods (0·03–0·1 sec.) at varied intervals, and the coincidence found between observed and actual interruptions indicates that there are no interruptions attributable to the falling of the image on a cluster of cones insensitive to the colour of the light. It is further shown that, irrespective of its colour, the image of the source moves over the same points of the retina, so that it cannot be supposed that, in the course of the small movements occurring during the "fixation" of the eye, red images move only over red-sensitive clusters and blue images only over blue-sensitive clusters. In another series of experiments, sources (0·3′) of different colours (red, yellow, and green) are flashed on for short periods of time (0·003–0·01 sec.), and the observer is required to name the colours of the flashes. The yellow flashes are never described as red or as green, whereas if there were red- and green-sensitive clusters on the fovea, some of the yellow flashes would be described as red, and some as green. These experiments prove the absence of clusters of diameter equal to or greater than that of the image of the source. It is shown also that an observer can see two small sources— one red and one green— as separate objects when the retinal images are only 1 μ. apart, i.e. less than the distance between cones, and he can correctly describe the relative positions of the sources.

A.E.S.

PATENTS

Creams containing Hair Dyes

Monsavon-l'Oréal *BP* 741,307

A cream containing a hair dye contains as its base the product obtained by treating a cationic surface-active base one of whose active basic groups contains S or N with an anion-active fatty acid, an acid sulphate of a fatty alcohol, or an alkylarylsulphonic acid, e.g. lauryldimethylsulphonium oleate. C.O.C.

Oxalate Coatings on Metal

Pyrene Co. *BP* 741,080

A continuous closely adherent coating is produced on a metal by treatment with an aqueous solution of oxalic acid containing titanium. Thus stainless steel immersed for 5 min. at 170°F. in a solution containing 0·2% oxalic acid, 5·0% NaCl, 0·5% sodium chlorate and 0·1% titanium (added as Ti oxalate) is given a firmly adherent dark green coating. C.O.C.

Colouring Mineral Surfacing Granules

Central Commercial Co. *USP* 2,700,003

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Copper-staining of Glass

Corning Glass Works *USP* 2,701,215

Ruby glass suitable for "Sealed Beam" lamps is produced by heating the glass in contact with a copper-staining composition in an atmosphere containing SO_2 and then heating the resulting yellow glass in a reducing atmosphere free of SO_2 until it becomes red. C.O.C.

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The article is formed, dried leather hard, sprayed with glaze and the dry, unmatured glaze then coated with a dispersion of a synthetic resin. The decoration is then applied to the resin surface by e.g. painting, screen printing or by use of a transfer. A single firing then suffices to mature the body, the glaze and the decoration. C.O.C.

Corrosion-resistant Treatment of Aluminium

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Producing Black Films on Ferrous Metals

A. F. Holden Co. *BP* 735,933

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Alexander Smith *USP* 2,706,143

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The lecture which was to have been given on Friday April 20th at the Waldorf Hotel, London

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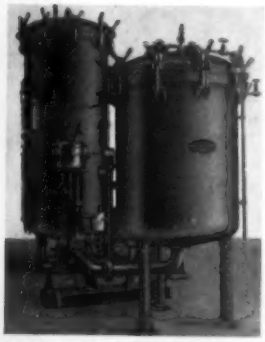

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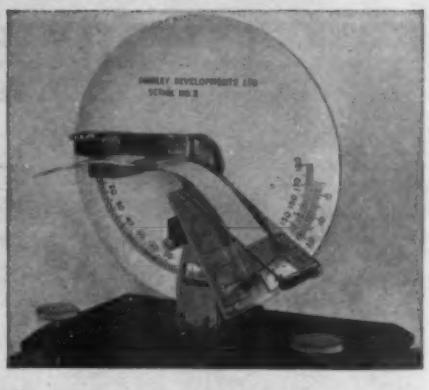

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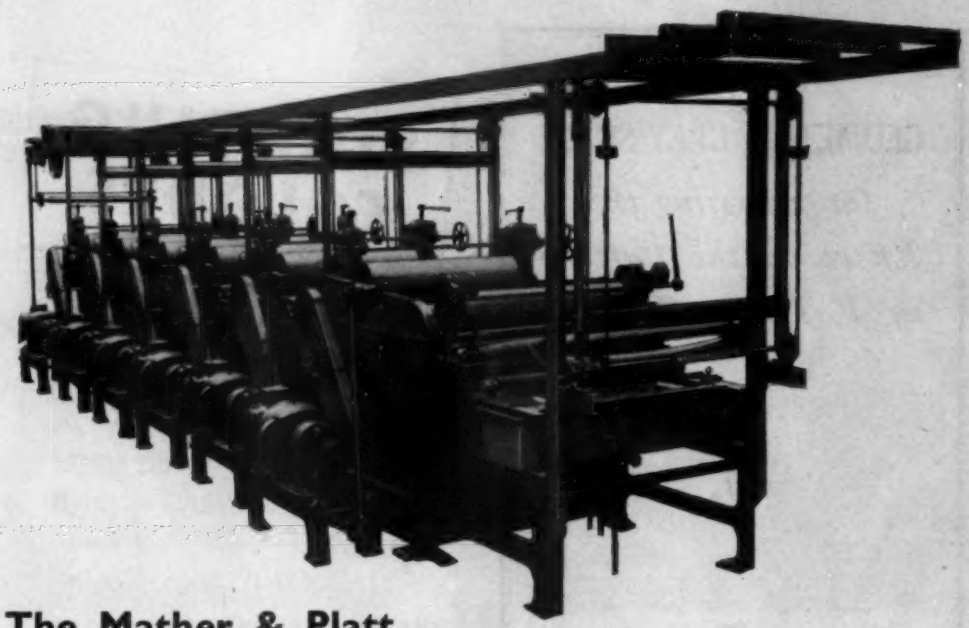

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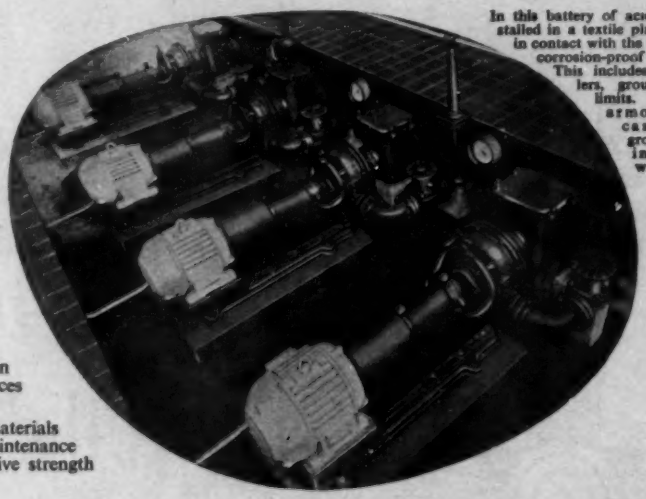

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